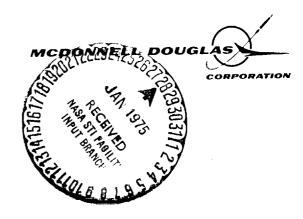


TEMPERATURE MATERIALS Yearly Summary Report (McDonnell-Douglas Astronautics Co.)

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CORROSION AND STRESS CORROSION SUSCEPTIBILITY OF SEVERAL HIGH TEMPERATURE MATERIALS

31 AUGUST 1972

MDC E0609

Yearly Summary Report

Submitted in Compliance with Contract Number NAS 8-27270

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MCDONNELL DOUGL

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FOREWORD

This Yearly Summary Report was prepared by McDonnell Douglas Astronautics

Company - East (MDAC-E) under NASA-MSFC Contract NAS-8-27270, Corrosion and Stress

Corrosion Susceptibility of Several High Temperature Alloys.

The program is essentially a two year effort that started on 19 May 1971 and is scheduled to end 19 May 1973. The program is under the direction of Mr. J. G. Williamson of the Metallic Materials Section, Materials Division of the George C. Marshall Space Flight Center, National Aeronautics and Space Administration.
Mr. J. W. Davis is the Program Study Manager for MDAC-E. Mr. R. M. Huffstutler supervised the laboratory diagnostic evaluation and mechanical properties testing.
Mr. K. C. Garland supervised the alternate immersion testing while Mr. M. B.
Munsell supervised the elevated temperature testing of the precracked specimens.
Mr. L. J. Pionke assisted in data analysis and preparation of the final report.

ABSTRACT

During the year the corrosion and stress corrosion susceptibility of six high temperature alloys, which were candidates for the thermal protection system on the Space Shuttle, was determined. The materials evaluated were: the nickel-base Inconel 718, Rene' 41, and Hastelloy X alloys, the cobalt-base L-605 alloy, the dispersion-strengthened Ni-20Cr-2ThO₂ material, and the silicide coated columbium alloy, FS-85. Precracked and stressed tensile specimens and unstressed control samples of each alloy were subjected to alternate immersion testing in 3.5 percent synthetic sea water. After 60 days exposure none of the alloys were found to be susceptible to any form of corrosion.

Specimens of each alloy were also subjected to simulated temperature/stress reentry profiles with and without salt coatings. After 50 cycles the salt coated samples exhibited no significant change in the creep, crack propagation, or tensile properties relative to the uncoated condition.

CORROSION OF HIGH TEMP ALLOYS

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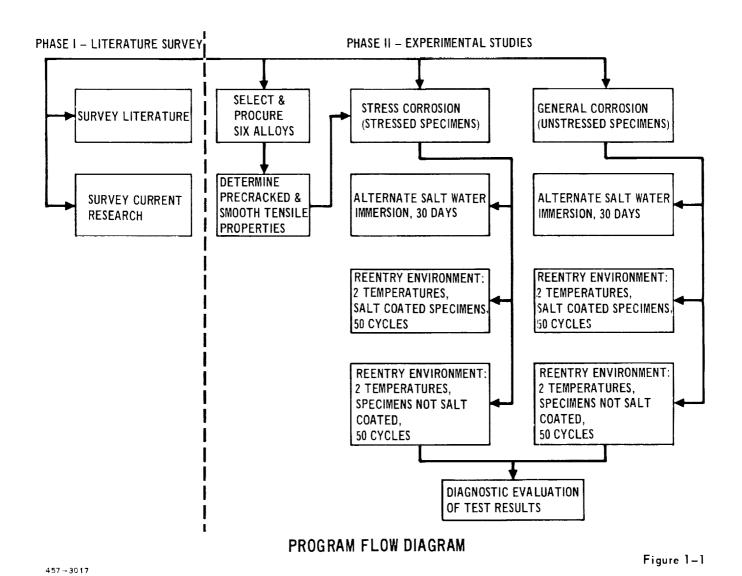
1.0 INTRODUCTION

Current plans for the Space Shuttle vehicle require that it be launched at ocean side facilities such as Cape Kennedy or Vandenberg AFB. A launch site near the ocean will impose potential corrosion conditions on the thermal protection system (TPS) of the vehicle if it is metallic. For example, on the ground between flights, the TPS will encounter atmospheric salt from the ocean and is likely to become salt coated. During the flight the aerodynamic heating will cause this salt deposit to become hot or molten. Such hot salt environments are known to cause stress-corrosion or severe general corrosion in some metals and their alloys. This program was initiated to determine if the candidate alloys for a metallic TPS were susceptible to this type of corrosion.

Two factors complicate the selection and characterization of metallic TPS material for such a space vehicle. One of these is the requirement for extensive reuse capability, which results in repeated loading through a complex high temperature-low-pressure-stress cycle and a long service life. This combination of cyclic loading, cyclic temperatures, and corrosive environment could promote short-time stress-corrosion failures in the TPS panels. The other factor is the thin sheet metal gages being considered for TPS panels (i.e., 0.010 to 0.020 inches thick); even minor pitting or slight intergranular corrosion of such thin material could reduce the net cross section to the point where premature failure becomes highly probable. Furthermore, such thin gages are generally joined by welding to construct stiffened skin TPS panels. Welding defects and faying surfaces in such panels are likely to add to a given stress corrosion problem.

The primary purpose of this program was to investigate the corrosion and stress corrosion susceptibility of candidate alloys for the shuttle TPS. The alloys selected for study were Inconel 718, Rene' 41, L-605, Hastelloy X,

Ni-20Cr-2ThO₂ (TDNiCr) and R512E coated FS-85. Both unstressed specimens and fatigue-precracked stressed specimens of each alloy were exposed to alternate immersion in an aqueous salt solution and to temperature-reduced-pressure-time cycles which closely simulated the anticipated shuttle operating conditions. In the latter tests, some samples were coated with salt to study corrosion effects, and others were tested without a salt coating. Use of two sets of specimens aided in distinguishing corrosion and stress-corrosion susceptibility from other environmental effects. The program organization is summarized in Figure 1-1.



1 - 2

2.0 PHASE I - LITERATURE SURVEY

At the start of this program, a literature survey was initiated to review past and present research directed toward determining the corrosion and stress corrosion of high temperature alloys, specifically, the nickel, cobalt, and columbium based alloys. Special emphasis was placed on studies which centered around exposing these alloys to elevated temperature. To accomplish this phase, the following sources were consulted:

- o A literature search, inclusive from 1962 to June 3, 1971, for references pertaining to corrosion and stress corrosion of superalloys and columbium obtained from the NASA Scientific and Technical Information Facility.
- o A report bibliography on superalloy and columbium corrosion and stress corrosion prepared by the Defense Documentation Center, Defense Supply Agency, Cameron Station, Virginia.
- o A report bibliography on superalloy and columbium corrosion and stress corrosion prepared by the Defense Metals Information Center, Battelle Memorial Institute, Columbus, Ohio.
- o Material vendors, research laboratories, airframe and jet turbine manufacturers, and others believed to be active in stress corrosion programs.

The extent of available data on the six alloys of this program is shown in Table 2-1. At lower temperatures, the behavior of nickel-base alloys Rene' 41, Inconel 718, TDNiCr, and cobalt-base alloy L-605 is documented. These alloys have been found to be immune to stress corrosion cracking (SCC) in room temperature tests such as alternate immersion or seashore exposure. The resistance of Rene' 41 and Inconel 718 to SCC at low temperature has been verified by several different investigators. Specific details are presented in Section 2.1, "Summary

of Previous Stress Corrosion Studies." Both Rene' 41 and Inconel 718 were considered candidates for service as skin materials for the SST, and their susceptibilities to SCC have been determined at temperatures up to 616°K (650°F). At this temperature neither alloy was found to be susceptible.

Table 2-1
SUMMARY OF AVAILABLE DATA ON THE CORROSION RESISTANCE OF HIGH
TEMPERATURE ALLOYS

	GENE	ERAL CORROSION	STRESS CORROSION				
ALLOY	SALT HOT WATER SALT		LOW TEMPERATURE	INTERMEDIATE TEMPERATURE	HIGH TEMPERATURE		
INCONEL 718 RENE' 41 L-605 HASTELLOY X TDNiCr FS85/R512E	NONE NONE NONE NONE NONE NONE	REF (9), (11) REF (10), (11), (12) REF (12) REF (10), (12) REF (11), (13) NONE	REF (1), (3), (4), (5) REF (1), (2), (5) REF (1) NONE REF (6) NONE	REF (3), (5) REF (2), (5) NONE NONE NONE NONE NONE	REF (11) REF (11) NONE NONE REF (11) REF (11)		

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The high temperature stress corrosion susceptibility of salt coated specimens of Inconel 718, Rene' 41, TDNiCr, and the R512E coated columbium alloy Cb-752 has been the subject of only one study, and the results indicate that severe degradation of the mechanical properties did occur. It was found that the extent of corrosive attack was independent of applied stress but extremely sensitive to the partial pressure of oxygen during the test.

Sulfidation, or hot corrosion, is a problem of great concern to gas turbine manufacturers, who wish to increase operating temperatures and service life in marine environments. Rene' 41, TDNiCr, Hastelloy X, and L-605 have been evaluated for hot corrosion resistance in jet-fueled burner rigs. All four alloys have been found to be susceptible in varying degrees to this sodium sulfate attack. Details are presented in Section 2.3.

2.1 <u>Summary of Previous Stress Corrosion Studies</u> - In a program to evaluate the chloride stress corrosion susceptibility of candidate alloys for the Supersonic Transport, Reference 1, the superalloys Rene' 41, Inconel 718, and L-605 were 2-2

evaluated. Test conditions consisted of three environments: 1000 hours in 5% salt spray, four seasons at the E1 Segundo Beach, and 500 hours alternate immersion in a synthetic sea salt solution. Test variables included sheet thickness (0.635 mm (.025 in.) and 1.27 mm (.05 in.)) and material conditions such as basic temper plus 1000 hours at 616°K (650°F), and grain direction. Fatigue precracked specimens were prepared from 1.27 mm (.050 in.) sheet in the basic-temper-plus-1000-hours-exposure at 616°K (650°F) and/or the welded condition.

Based on the results of this investigation, it was found that the superalloys studied were immune to stress corrosion cracking. None of the superalloys failed during stress corrosion testing regardless of temper, welding, or surface preparation; nor did they show reduction of mechanical properties attributed to stress corrosion. Tables 2-2, 2-3, and 2-4 show the resistance of these meterials in alternate immersion, 5% salt spray, and beach exposure, respectively.

In an evaluation of the effects of protective coatings on the stress corrosion properties of SST skin materials (Reference 2), eleven combinations of coatings and substrates plus bare metals were studied.

Rene' 41 was found to be resistant to stress corrosion attack from salt either in the hot condition at 286°K (55°F) or the humid condition at 308°K (95°F). This conclusion was based upon exposures of up to 7000 hours of stressed, salt coated specimens in either a saturated-humidity chamber at 308°K or a circulating-air electric oven at 561°K . The specimens were prepared from sheet material, self-stressed to $1030~\text{MN/m}^2$ (150 ksi) by double-bowing, and salt-coated by hand dipping in a 20% aqueous salt solution.

Under a NASA-MSFC contract to develop an accelerated stress corrosion test for ferrous and nickel alloys (Reference 3), eight alloys, including Inconel 718 were exposed to four laboratory environments: 3.5% NaCl and 339% (150%), 20% NaCl at 339%, 42% MgCl $_2$ at 377% (220%), and to natural exposure in seacoast

			NET SE APPLIED			RESIDUAL NOTCHED TENSILE STRENGTH	
ALLOY	CONDITION	MN/m ²	KSI	PERCENT OF NOTCHED ULTIMATE STRENGTH	HOURS TO FAILURE	MN/m ²	KSI
INCONEL 718	AGED* WELDED	1040 570	150 82.6	80.0 80.4	NF** NF**	1310 792	190 115
L-605 (20% COLD ROLLED)	AGED* WELDED	877 639	127 92.6	79.5 81.9	NF** NF**	116 0 775	168 112
RENE' 41 (20% COLD ROLLED AND AGED***)	AGED* WELDED	1140 756	165 109	80.0 80.5	NF** NF**	1420 875	206 127

NOTE: ALL DATA OBTAINED USING TENSILE SPECIMENS FROM 0.127 mm (0.050 IN.) SHEETS, PRECRACKED AND EXPOSED TO ALTERNATE IMMERSION IN SYNTHETIC SEA WATER FOR 500 HOURS UNDER SUSTAINED APPLIED STRESS.

- *AGED FOR 1.000 HOURS AT 616°K (650°F)
- **NO FAILURE AFTER 500 HOURS
- ***AGED FOR 16 HOURS AT 10330K (14000F)

Table 2-3
RESULTS OF SALT SPRAY EXPOSURE (REFERENCE 1)

	CONDITION			NET SECT PPLIED ST		RESIDUAL NOTCHED TENSILE STRENGTH		RESIDUAL Notched	
ALLOY		SPECIMEN ORIENTATION	MN m ²	KSI	PERCENT OF SMOOTH			TENSILE ELONGATION	
					F _{ty}	MM m ²	KSI	%, IN 2.54 CM (1.0 IN)	
INCONEL 718	AGED + 1000 HOURS AT 616°K (650°F)	TRANSVERSE	1080	156	90	1420	206	18,9	
	BRAZE CYCLE HEAT TREAT THEN WELDED	TRANSVERSE	661	95,9	90	980	142	6.0	
L-605 (20% COLD	1000 HOURS AT 616 ⁰ K (650 ⁰ F)	TRANSVERSE	716	103	90	1220	177	19,7	
ROLLED)	AS WELDED	LONGITUDINAL	706	102	90	1080	157	11.4	
RENE' 41 (20% COLD	AGED 16 HOURS AT 1033 ⁰ K (1400 ⁰ F)	TRANSVERSE	1250	182	90	1560	226	11.5	
ROLLED)	1000 HOURS AT 616 ⁰ K (650 ⁰ F)	TRANSVERSE	1250	182	90	1540	224	11.0	
	BRAZE CYCLE HEAT TREAT	TRANSVERSE	1220	177	90	1560	226	12.5	
	SURFACE PREPARATION	TRANSVERSE	1260	183	90	1550	225	9,1	
	WELDED THEN AGED	LONGITUDINAL	1230	179	90	1510	219	5.5	

NOTE: ALL DATA OBTAINED USING TENSILE SPECIMENS MACHINED FROM 0,127 mm (0,050 INCH) SHEET, PRECRACKED AND EXPOSED TO 5% SALT SPRAY FOR 1000 HOURS UNDER A SUSTAINED APPLIED STRESS.

Table 2-4

RESULTS OF NATURAL EXPOSURE FOR 20% COLD ROLLED RENE' 41 (REFERENCE 1)

CONDITION	NET SECTION APPLIED STRESS MM/m ² KSI		PERCENT OF SMOOTH F _{ty}	RESIDUAL TENSILE S MN/m ²		RESIDUAL NOT CHED TENSILE ELONGATION, %, IN 2.54 CM (1.00 IN)		
AGED 16 HR AT 1033 ⁰ K (1400 ⁰ F)	1050 1250	152 182	75 90	1600 1600	232 232	8.4 8.8		
AGED 16 HR AT 1033°K (1400°F) PLUS 1000 HR AT 616°K (650°F)	1050 1250	152 182	75 90	1610 1620	233 235	10.3 10.8		
WELDED THEN AGED 16 HR AT 1033°K (1400°F)	1030 1230	149 179	75 90	1510 1560	218 226	5.0 4.3		

NOTE: ALL DATA OBTAINED USING TRANSVERSE TENSILE SPECIMENS MACHINED FROM 0.127 mm (0.050 IN.) SHEET,
PRECRACKED AND EXPOSED FOR ONE YEAR AT A DISTANCE OF 91.4 METERS (300 FT) FROM THE MEAN TIDE
LINE AT EL SEGUNDO BEACH, CALIFORNIA, UNDER A SUSTAINED APPLIED STRESS.

test racks. Single edge-notched and fatigue precracked specimens were used to eliminate the excessively long crack initiation period, and to permit the use of a fracture mechanics approach. Laboratory specimens were loaded under plane strain conditions and placed in the salt solutions to determine $K_{\rm Iscc}$, the stress intensity factor for stress corrosion. Identical specimens were tested in air to determine $K_{\rm Ix}$, the stress intensity factor at maximum load in the absence of corrodent. This measure of fracture toughness was used instead of the more common $K_{\rm Ic}$ (i.e., the stress intensity factor at the point of plane strain failure, or more commonly, at "pop-in") because of the difficulty in detecting the pop-in load in some alloys.

The data were plotted in terms of a stress intensity ratio ($K_{\rm Iscc}/K_{\rm Ix}$) versus failure time. For the Inconel 718 specimens that underwent seacoast exposure, this ratio was found to be greater than 0.869. Based on this study it was concluded that Inconel 718 exhibited excellent seacoast corrosion resistance and was immune to stress corrosion in 3.5 and 20 percent NaCl solutions at room tempera-

ture and 339°K (150°F). However, comparatively rapid stress corrosion occurred in 42 percent MgCl $_2$ at 377°K (220°F).

In an in-house corrosion study conducted at Marshall Space Flight Center, Reference 4, Inconel 718 bar specimens were subjected to alternate immersion in a 3.5 percent solution of NaCl. The stress levels varied from 241 MN/m^2 (35 ksi) to 1210 MN/m^2 (175 ksi) in the transverse direction and 710 MN/m^2 (103 ksi) in the longitudinal direction, representing 25 to 100% yield stress and 75% yield stress, respectively. No failures occurred during the six month testing period. Both 19.0 mm (.75 in.) and 25.4 mm (1.00 in.) aged bar stock were evaluated.

In another study involving the evaluation of six candidate materials for the SST, Reference 5, cantilever-type sheet specimens of Rene' 41 and Inconel 718 were stressed from 23 to 90 percent of the 0.2 percent yield strength and exposed to two environments: cyclic exposure at 616° K $(650^{\circ}$ F) in circulating air, and sustained exposure in a humidity cabinet at 298° K $(77^{\circ}$ F). Surface treatments included braze coating, salt coating $(6 \text{ parts NaCl} \text{ and } 1 \text{ part MgCl}_2)$, and braze plus salt coating. The braze alloy selected was Premabraze 130 (72 w/o Au, 6 w/o Cr, and 22 w/o Ni). The stress levels to which the specimens were subjected are listed in Table 2-5.

Table 2-5

MAXIMUM STRESS LEVELS APPLIED TO CANTILEVERED TEST SPECIMENS IN REFERENCE (5)

		NOTCHED		UNNOTCHED			
ALLOY	MAXIMUM STRESS		% F _{ty}	MAXIMUN	% F _{ty}		
	MN/m ²	KSI	ty	MN/m ²	KSI	ty	
RENE' 41	874	127	85.6	337	48.8	33.0	
INCONEL 718	1020 637 727	147 92.4 106	100.0 65.5 74.8	396 235 282	57.4 34.0 40.8	38.9 24.1 28.9	

NOTE: VARIATIONS BETWEEN SPECIMENS STRESS LEVELS ARE DUE TO DIFFERENCES IN THE FREE LENGTH OF THE CANTILEVER ARM.

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After 20,000 hours of a 30,000 hour test, it was found that neither Rene' 41 nor Inconel 718 was susceptible to stress corrosion cracking in the presence of chloride salt at 616°K (650°F) and stress levels above 25% yield stress.

The stress corrosion susceptibility of TDNiCr was studied as part of a comprehensive program to develop dispersion-strengthened metal structures,

Reference 6. Sheet specimens, 0.51 mm (0.020 in.) thick, were exposed to alternate immersion in a 3.5 percent salt solution and natural exposure in a test rack, fifty meters from the Pacific Ocean at El Segundo Beach. In both tests specimens were prestressed to 90% of their 0.2 percent yield strength. No failures occurred during the 500 hours alternate immersion testing. No surface attack was visible, and only a light residue was observed on the specimens. After the alternate immersion tests, the same specimens were examined and then further tested at the beach. No failures occurred after 1420 hours of beach exposure, but some surface pitting had occurred. The beach exposure was considered to be more corrosive than alternate immersion. Post-test tensile strength is presented in Table 2-6.

Table 2-6

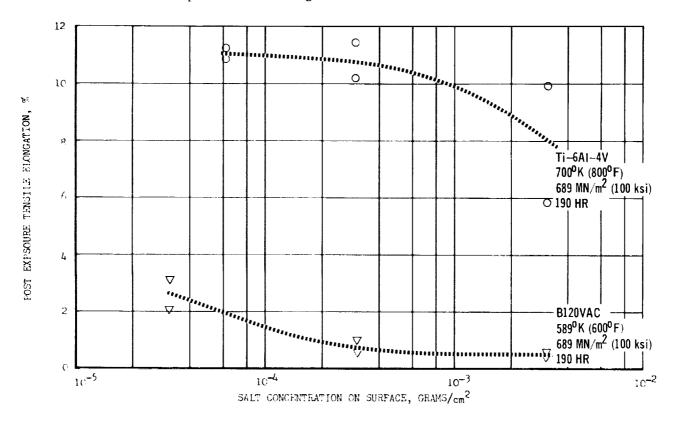
ROOM TEMPERATURE TENSILE PROPERTIES OF TDNiCr BEFORE
AND AFTER SALT WATER EXPOSURE (REFERENCE 6)

	NO EX	POSURE		AFTER EXPOSURE*					
F _{ty}		F _{tu}		F,	ty	F _{tu}			
MN/m ²	KSI	MN/m ²	KSI	MN/m ²	KSI	MN/m ²	KSI		
569 577	82.5 83.6	868 877	126 127	563 ' 585	81.6 84.8	793 718	115 104		

^{*500} HOURS ALTERNATE IMMERSION IN 3½% SALT SOLUTION FOLLOWED BY 1420 HOURS EXPOSURE AT EL SEGUNDO BEACH, 90% YIELD STRESS MAINTAINED DURING TESTING.

2.2 <u>Effects of Salt Concentration</u> - Based on the results of the literature survey it was determined that the amount of salt deposited on a specimen has a direct bearing on the stress corrosion susceptibility of some materials. For

example, in a program to study the effects of alkali-halide corrodents on the stress corrosion cracking of titanium alloys, Reference 7, it was found that the titanium alloys exhibited less ductility upon post-exposure tensile testing as surface concentration of salt is increased. The percent elongation of tensile specimens after 190 hours exposure at intermediate temperatures as a function of salt concentration is presented in Figure 2-1.



EFFECT OF SURFACE SALT CONCENTRATION ON THE TENSILE ELONGATION OF SPECIMENS AFTER 190 HOURS ELEVATED TEMPERATURE EXPOSURE (REFERENCE 7)

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In another literature survey conducted in Reference 1 program, thirteen organizations involved in corrosion research were contacted. Of those thirteen companies, three commented that the concentration of salt on the specimens had a significant effect on the corrosion susceptibility of these materials. The most significant comment came from the National Bureau of Standards which stated that the results of natural exposure tests run at Kure Beach, North Carolina, were

dependent upon the distance from the test rack to the mean low tide. The results of these tests for two different steels are shown in Table 2-7.

Table 2-7
STRESS CORROSION FAILURES IN MARINE ATMOSPHERE AT KURE BEACH, N.C.
(REFERENCE 1)

DISTANO	DISTANCE FROM MEAN TIDE LINE		AM-350 ST	TEEL	AM-355 STEEL			
MEAN T			STRESS		STR	ESS	DAYS TO	
METERS	FT	MN/m²	KSI	DAYS TO FAILURE	MN/m ²	KSI	FAILURE	
24.4	80	821*	119	17 17 17 20 22	852*	124	3 3 3	
244.0	800	821*	119	26 26 38 47	852*	124	18 18 19	

*75% OF 0.2% YIELD STRENGTH

NOTE: HEAT TREATMENT IS 2000K (-1000F) FOR 3 HOURS, PLUS 7270K (8500F) FOR 3 HOURS

In Reference 8 it was also found that thicker deposits of salts increase the corrosion rates of Inconel 702 and degrade the mechanical properties. This change in properties is reflected in the data presented in Table 2-8. The data is based upon 0.355 mm (.015 in.) sheet coated with lithium fluoride or potassium chloride and tested at 1311°K (1900°F) under a stress of 17.24 MN/m² (2500 psi). The time to rupture decreased as the LiF weight increased, leveling off at the higher weights.

Recent data on the effect of salt concentration on the properties of Inconel 718 were generated at Marshall Space Flight Center, NASA, Reference 9. Tensile specimens were coated with varying thicknesses of sodium chloride and then exposed to 978°K (1300°F) for 48 hours. The specimens were not stressed. The effect of the salt concentrations on the residual properties of Inconel 718 are presented in Table 2-9. It can be seen from this data that the thicker the salt coating the greater the damage to the specimen. 2-9

Table 2-8

EFFECT OF SALT THICKNESS ON CREEP - RUPTURE TIME FOR INCONEL 702* (REFERENCE 8)

WEIGHT OF LiF	TIME TO RUPTURE	WEIGHT OF KC1	TIME TO RUPTURE		
(mg/cm ²)	(HR)	(mg/cm ²)	(HR)		
0.0	DISC**	0.0	DISC** DISC** 8.7		
0.18	DISC**	0.16			
0.29	2.4	0.64			
0.60	1.8	1.1	7.7		
1.1	1.7	1.5	4.6		
1.5	2.0	2.1	2.7		
2.2	1.4	3.6	3.2		
4.7	0.8	10.2	5.0		
13.1	0.9				

^{*}TESTING CONDITIONS: 1311°K (1900°F) UNDER A STRESS OF 17.2 MN/m²

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Table 2-9

EFFECT OF SALT THICKNESS ON RESIDUAL PROPERTIES OF INCONEL 718 (REFERENCE 9)

	TEMPERATURE		SALT TH	SALT THICKNESS		F _{ty}		tu	ELONGATION,
ĺ	οK	°F	mm	IN.	MN/m ²	ksi	MN/m ²	ksi	PERCENT
ĺ	298	75	NONE		986	143	1365	198	19.5
١	977	1300	l no	NE	979	142	1289	187	19.5
١	977	1300	0.0051	0.0002	972	141	1241	180	17.5
١	977	1300	0.0127	0.0005	965	140	1134	179	10.0
١	977	1300	0.0737	0.0029	807	117	903	131	1.5
	977	1300	0.4369	0.0172	_	_	431	62.5	0.5

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2.3 Hot Salt Corrosion - Rene' 41 and Hastelloy X were among ten alloys evaluated under an Air Force program, Reference 10, to determine the effects of welding and brazing on the properties of salt coated and bare tensile specimens having thicknesses ranging from 1.42 mm to 2.35 mm (.056 to .093 inch). Unnotched tensile specimens were used in the study, with 6.35 mm (.250 inch) long brazed or welded layers located in the central portion of the 4.8 cm (1.9 inch) long gage length. The Hastelloy X brazed specimens were prepared using braze alloy AMS 4776 while the Rene' 41 specimens were prepared with the J8600 braze alloy; in either case, a brazing temperature of 1450°K (2150°F) was used. Simulated sea salt containing

^{**}TEST DISCONTINUED AT 30 HOURS WITHOUT FAILURE

48.1% NaCl, 21.2% MgCl_2 · $6\mathrm{H}_2\mathrm{O}$, 7.7% $\mathrm{Na}_2\mathrm{SO}_4$, and 23% CaCl_2 was brushed onto the gage section of each specimen until a nominal thickness of .05 mm (.002 in.) was obtained. Both salt coated and bare specimens were subsequently thermal cycled under a constant applied stress for up to 63 cycles. One thermal cycle was defined as three hours at temperature and an hour heat-up and cool-down. Stress levels and temperatures for the two alloys of interest are listed in Table 2-10.

Table 2-10
TEST CONDITIONS FOR HOT SALT CORROSION TESTING OF HASTELLOY-X AND RENE' 41 (REFERENCE 10)

		STRESS	LEVEL A	T TEMPE	RATURE	
MATERIAL	1144 ⁰ K (1	600 ^o F)	1255 ⁰ K	(1800°F)	1366 ⁰ K (2000 ⁰ F)
	MN/m ²	KSI	MM/m ²	KSI	MN/m ²	KSI
HASTELLOY-X	24.1	3,5	NO	NE	13.8	2.0
	20.7	3.0			6.9	1.0
RENE' 41	117	17.0	27.6	4.0	NOI	NE.
	89.2	13.0	20.7	3.0		

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No evidence of salt corrosion was found on welded specimens of Hastelloy X, exposed at 1144°K (1600°F) nor was there any significant difference in tensile strengths. At 1366°K (2000°F), however, seven out of eight specimens failed before completing their schedules, the failures being typical ductile stress-rupture breaks. No evidence of corrosion was present, and the authors concluded that the temperature/stress levels were too severe for this alloy.

Salt was found to decrease the ultimate strength and ductility of brazed Hastelloy X both at 1144°K and 1366°K. Such behavior was attributed to more extensive surface cracking of the salt coated specimens. Failures occurred in the brazed region of salted specimens and outside the brazed region of unsalted specimens.

A degradation of tensile properties was observed for both welded and brazed specimens of Rene' 41; subsequent metallographic examination showed that all

specimens had over-aged during their respective thermal exposures. Severe galvanic corrosion was observed for the brazed specimens, whereas only minor surface cracking occurred in the welded specimens.

Based on this study it was concluded that both alloys were more susceptible to salt corrosion in the brazed form than in the welded form. Of the ten alloys studied, Rene' 41 was found to be one of the two materials most susceptible to salt corrosion in the brazed condition.

Only one study (Reference 11) has been published to evaluate the hot salt corrosion and stress corrosion of alloys to be used for metallic heat shields. The materials evaluated included Inconel 718, Rene' 41, TDNiCr, and Cb752/R512E. The stress corrosion tests were conducted using both unnotched tensile and precracked double cantilever beam specimens with a nominal thickness of 0.50 mm (.020 in.). The specimens were exposed for 48 hours to elevated temperatures in both the salt coated and bare conditions and tested to determine residual mechanical properties. The salt coating was applied to the gage section of the specimens in the form of a slurry approximately 0.6 mm (.015 in.) thick. To obtain information on the effect of oxygen pressure on hot salt corrosion, specimens were exposed in air, in a mixture of 1% oxygen - 99% argon, and in pure argon, all at a total pressure of one atmosphere.

The results of the tensile testing, shown in Table 2-11, indicate that the mechanical properties of all salt coated specimens are severely degraded in the air environment. The elongation to failure after exposure to salt and air was reduced to near zero in most cases. Whereas stress generally had little influence on the residual properties, oxygen partial pressure had a significant effect in that the degradation was considerably less in the 1% oxygen environment and absent altogether in the inert environment.

Crack propagation tests were performed on precracked double cantilever beam specimens of Inconel 718, Rene' 41, and Cb752/R512E after elevated temperature 2-12

EFFECT OF ELEVATED TEMPERATURE EXPOSURE ON THE TENSILE PROPERTIES OF SALT COATED HIGH TEMPERATURE ALLOYS (REFERENCE 11)

	BYDORINGE	101		HAPOCIBE	181		VIELD	٥	TIMATE	1	
\ \ \ \	TEMPEDATIBE	TIBE	T 142	CTET	מ נו	FNYIDONMENT	CTPE	ץ פ	CTPEC	ر د	EL ONCATION
	λ γ	- -	5	MN/m^2	SS S		MN/m ²	KSI	MN/m ²	KSI	(PERCENT)
INCONEL 718	716	1300	ON.	1	ı	UNEXPOSED	1370	198	1500	218	18.6
			욷	55.2	∞	AIR-OXIDIZED	965	140	1060	154	25
			9	0	0	AIR-OXIDIZED	1030	150	1280	185	22.6
			YES	65.5	9.5	ARGON	1100	160	1420	506	21
			YES	0	0	ARGON	1100	160	1450	210	20
			YES	29.0	4.2	1% 0 ₂ – 99% Ar	965	140	1090	158	3,3
			YES	0	0	1% 02 - 99% Ar	965	140	1170	170	&
			YES	55,2	∞	AIR-OXIDIZED	827	120	331	135	2
			YES	•	0	AIR-OXIDIZED	834	121	910	132	3,3
			YES	65.5	9.5	AIR-NO OXIDE	069	901	1020	148	4.6
			YES	0	0	AIR-NO OXIDE	1	1	225	80	1
RENE' 41	1144	1600	ON.	1	1	UNEXPOSED	634	92	1100	160	23
			9	20.7	က	AIR	910	132	1030	149	9.6
			<u>Q</u>	0	0	AIR	883	128	1070	155	22.6
			YES	20.7	ж	AIR	483	70	655	95	1.7
			YES	0	0	AIR	455	99	621	8	2.6
TDNiCr	1322	1920	NO	ı	1	UNEXPOSED	292	82	814	118	14.6
			Q.	4.14	9.0	AIR	238	78	6//	113	13.5
			2	0	0	AIR	469	88	807	117	33
			YES	11.7	1.7	ARGON	238	82	821	119	14
			YES	0	0	ARGON	225	8	814	118	19
			YES	0	10.6	$1\% 0_2 - 99\%$ Ar	*	*	*	*	*
			YES	0	0	$1\% 0_2 - 99\% \text{ Ar}$	441	42	069	100	25.4
			YES	4.1	9.0	AIR	214	31	317	46	1.0
			YES	0	0	AIR	44]	64	724	105	12
			YES	11.7	1.7	AIR PRE-OXIDIZED	1	1	241	33	~
			YES	٥		AIR PRE-OXIDIZED	1	'	193	28	
Cb752/R512E	1322	1920	Q.	ı	i	UNEXPOSED	414	93	231	11	21
			ON N	4.1	9.0	AIR	44	25	231	11	22
			<u>Q</u>	•	0	AIR	414	33	238	78	19
			YES	4.1	9.0	AIR	427	23	224	9/	10
			YES	0	0	AIR	441	2	238	78	18
	1477	2200	£	4.1	9.0	AIR	414	33	538	78	23
			2	•	0	AIR	414	8	524	9/	19
			YES	4.1	9.0	AIR	421	15	421	61	0
			YES	0	0	AIR	414	93	510	74	6
* * PDOKE DIEDING EVENCIEDE	Tangoring C										

NOTE: DATA OBTAINED FROM ROOM TEMPERATURE TESTS OF 0.05 mm (0.020 IN.) THICK SMOOTH TENSILE SPECIMENS AFTER 48 HOURS CONTINUOUS EXPOSURE TO THE TEMPERATURE INDICATED. 457-3029 *BROKE DURING EXPOSURE.

exposure. A drop in the critical stress intensity for crack propagation was observed for coated specimens of Rene' 41 and Cb752/R512E relative to the uncoated condition. Incomel 718 was found to be virtually unaffected.

In an attempt to elucidate the mechanism of degradation, coupons of Inconel 718, Rene' 41 and TDNiCr were subjected to 24 hour immersion in NaCl held at 1093°K (1500°F). The test results indicated that the corrosion rates were highly dependent on oxygen pressure and to some extent on the water content of the salt. Analysis of the sodium content of all alloys indicated that diffusion of sodium into the base metal had occurred.

The corrosion resistance of Rene' 41, Hastelloy X, and cobalt-base alloy L-605 to diesel fuel combustion products containing small concentrations of sea salt was evaluated as part of a program (Reference 12) to develop new hot-corrosion resistant alloys for marine gas turbines. Specimens in the form of cast pins, 3.17 mm (.125 in.) in diameter were tested in combusted diesel fuel containing one percent sulfur and 5 ppm sea salts for up to 1000 hours, with thermal cycling to room temperature every 50 hours. Surface loss and maximum penetration for each alloy of interest is listed in Table 2-12.

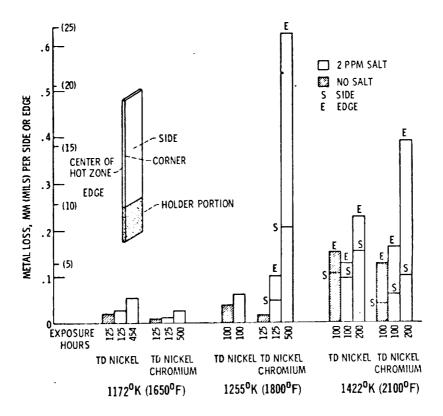
Table 2-12

HOT CORROSION BEHAVIOR OF SUPERALLOYS IN COMBUSTED DIESEL FUEL WITH ADDED SALT (REFERENCE 12)

	TEMPER	ATURE	SURFA	CE LOSS	MAXIMUM PI	NETRATION
	οK	°F	mm	10 ⁻³ IN.	mm	10 ⁻³ IN.
RENE´41	1144	1600	0.006	2.6	0.262	10.3
	1255	1800	0.015	6.8	0.780	30.8
HASTELLOY X	1227	1750	0.046	1.8	0.305	12.0
	1255	1800	0.050	1.8	0.290	11.3
L-605	1227	1750	0.140	5.5	0.390	15.3
	1255	1800	0.330	1.3	0.390	15.2
	1311	1900	0.830	32.7	1.060	41.9

^{*}TESTED AT 5 ppm SEA SALT, 500 HOURS; ALL OTHERS, 1000 HOURS.

A similar program, Reference 13, evaluated the corrosion resistance of TDNi and TDNiCr in an environment consisting of a Mach 0.5 gas stream of A-1 jet fuel combustion products containing 2 ppm simulated sea salt. Sheet specimens with a thickness of 1.52 mm (.060 in.) were cycled up to 500 times between room temperature and test temperatures of 1172°K (1650°F), 1255°K (1800°F), or 1422°K (2100°F). Hot zone metal losses for each test condition is shown in Figure 2-2. Both alloys were found to be susceptible to hot corrosion attack at 1172°K (1650°F) and at 1255°K (1800°F). No evidence of hot corrosion was found after the 1422°K (2100°F) testing although TDNiCr lost 0.10 mm (4 mils) per side by oxidation. Edge attack was exaggerated in all cases and was attributed to a geometrical effect.



TEST CYCLE: 1 HOUR AT TEST TEMPERATURE, 3 MINUTE FORCED AIR, COOL TO ROOM TEMPERATURE.

HOT ZONE METAL LOSSES FOR TDNI AND TDNICT RESULTING FROM CYCLIC EXPOSURE IN SIMULATED ENGINE ENVIRONMENT (REFERENCE 13)

Figure 2-2

457-3031

2.4 <u>Survey of Current Research</u> - A complete list of individuals contacted for the current research survey is included in Appendix A. Of those organizations polled, only the Space Division of North American Rockwell reported that they were involved in research on the stress corrosion susceptibility of high temperature alloys. The results of this investigation were discussed above, Reference 11.

Of the other organizations contacted, some referenced related reports and papers and the remainder could provide no information. Sherritt Gordon Mines, Ltd., submitted an AFML report covering their recent work to improve the oxidation resistance of dispersion strengthened nickel-chromium alloys, the Marquart Company submitted a report of alternate immersion testing of steels, and the International Nickel Company submitted two papers on hot corrosion. General Electric recommended that we consult References 1, 14 and 15. Westinghouse Research and Development Center is working on a contract from the Department of the Interior to develop a low cost iron base alloy to resist corrosion in hot sea water.

2.5 Conclusions:

- o Nickel-base superalloys Inconel 718, Rene' 41, Hastelloy X, and TDNiCr, and cobalt-base superalloy L-605 are apparently not susceptible to stress corrosion by sea salt at low temperatures.
- o The nickel base Inconel 718, Rene' 41 and TDNiCr and the R512E coated columbium alloy Cb-752 are susceptible to hot salt corrosion at elevated temperatures in air environments.
- o No published studies of stress corrosion susceptibility of L-605 have been made.
- No stress corrosion studies in simulated reentry conditions have been reported.
- o Hastelloy X is apparently not corroded by sea salt at 1144°K (1600°F)

and 1366°K (2000°F) in the welded form, but is attacked in the brazed form. Brazed Rene' 41 is susceptible to salt corrosion at 1144°K (1600°F) and 1255°K (1800°F).

At the time this program was initiated, the literature survey indicated that all stress corrosion studies on the alloys of interest had been carried out at low temperatures. The study cited in Reference 11 had only begun and little information on the testing conditions was available. The main objective of this program was to confirm the results of previous alternate immersion testing on the alloys of interest and to investigate their previously unknown stress corrosion susceptibility under cyclic time/elevated temperature/reduced pressure conditions that simulated those encountered by space vehicle upon reentry.

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3.0 TEST SPECIMENS

3.1 <u>Materials</u> - Because of weight considerations, thin gages would be used for a metallic thermal protection system (TPS). Present studies indicate that sheet metal thickness in the range of .254 mm (.010 inch) to .508 mm (.020 inch) are being considered for this application. Because these thin gages magnify the concern for general corrosion, .254 mm (.010 inch) thick sheet material was selected for study under this contract.

The materials evaluated under this program were Rene' 41, Inconel 718, Hastelloy X, Ni-20Cr-2ThO₂ (TDNiCr), L-605 (Haynes 25) and the R512E coated columbium alloy FS-85. To insure that the material used would be representative of current manufacturing technology, all of the material was procured to applicable specifications and the material suppliers were required to submit certified test reports covering chemistry and mechanical properties. A summary of the supplier's certification is presented in Table 3-1.

The use of superalloys and coated columbium for a metallic TPS would be as radiative heat shields, which must have an emittance high enough for the efficient reradiation of aerodynamic heat. For superalloys, a high emittance is usually provided by the material's oxide. This oxide is obtained by heating the material in the air to an elevated temperature and air cooling after a short holding period. For this program, the heat oxidation process was combined with the metallurgical heat treatments required for strength in the respective alloys. The heat oxidation temperatures used for each alloy are summarized in Table 3-2. In the case of the FS-85 columbium alloy, no additional thermal treatment was necessary because the fused slurry silicide R512E coating provided not only oxidation resistance but also high emittance.

Table 3-1
SUMMARY OF SUPPLIERS CERTIFICATION

INCONEL 718	RENE' 41	DACTELL OF T		1	1	_
	KEAR AT	HASTELLOY X	TDNiCr	L605	FS-85	
Techalloy	Universal Cyclops	Rodney Metals	Fansteel	Universal Cyclops	Fanstee1	
HT74B5ES	L1135K2	2600-0-4450	TD 3419	C10130	85D-3009	
.04	.077	.09	.025	.12	0053	
-	-	-	_		1	
_	-	_	_	-		
60	1 /0	-	-			
		_	_	-	_	
			19.76	19.89	_	
		1	-	2.33	.005	
			-	1.48	-	
-	9.02		_	-	_	
.007	-006	1	_		-	
· ·		1	.0038		-	- 1
-	• • • •	.40	-	.29		-
.93	3.24	_	1 _	_		١
-	-	30	_	14.60		١
-	-	-	_	14.08		1
5.16	-	_	_	_	1.0	١
-	-	_	2.02	_		١
、-	-	-	_ ,			1
		2.0	_	Ba1	Da1	1
Bal	Bal	Bal	Bal	10.02	_	
_	137.6	12/. 5	101.0	3165		
_						
- 1						
_			20	j.		l
	_	O	_	5	8	
AMS 5596 Chemistry Only	AMS 5545* Chemistry Only Solution Treated at 1394 OK	AMS 5536	MMS 1503	AMS 5537	Fansteel \$82402	
	.0460 18.08 18.59 .10 2.94007 .20935.1603 Bal	Techalloy Universal Cyclops HT74B5ES L1135K2 .04 .077	Techalloy Universal Cyclops Metals HT74B5ES L1135K2 2600-0-445G .04 .077 .09	Techalloy Universal Cyclops Metals Fansteel Cyclops Metals HT74B5ES L1135K2 2600-0-4450 TD 3419 .04 .077 .09 .025	Techalloy Universal Cyclops Metals Fansteel Universal Cyclops HT74B5ES L1135K2 2600-0-4450 TD 3419 C10130 .04 .077 .09 .025 .12	Techalloy Universal Cyclops Metals Fansteel Universal Cyclops Metals Cyclops HT74B5ES L1135K2 2600-0-4450 TD 3419 C10130 85D-3009 .04

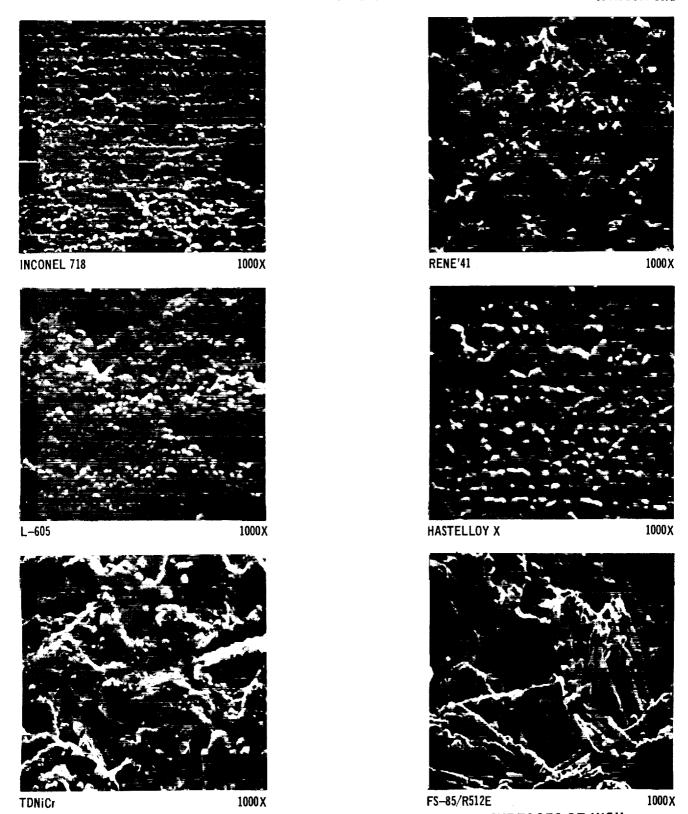
Table 3-2
EMISSIVITY/HEAT TREATMENT FOR HIGH TEMPERATURE ALLOYS

ALLOY	FINAL EMISSIVITY/HEAT TREATMENT
INCONEL 718	1241°K (1775°F) – 1/2 HOUR + 991°K (1325°F) – 8 HOURS + 894°K (1150°F) – 10 HOURS
RENE´41	1394 ⁰ K (2050 ⁰ F) - 1/2 HOUR + 1172 ⁰ K (1650 ⁰ F) - 4 HOURS
L-605	1352°K (1975°F) – 1/2 HOUR
HASTELLOY X	1352 ^o K (1975 ^o F) - 1/2 HOUR
TDNiCr	1449 ^o K (2150 ^o F) – 1 HOUR
FS-85	SILICIDE COATED AT 1689°K (2580°F) FOR 1 HOUR IN VACUUM

NOTE: ALL HEAT TREATMENTS WERE CARRIED IN AN AIR ATMOSPHERE EXCEPT WHERE NOTED.

Scanning Electron Microscope photographs of the surface of each alloy after their respective final heat treatments are shown in Figure 3-1. The surface roughness of the R512E coated FS-85 alloy evident in these photographs is typical of silicide coatings.

The microstructures that occur as a result of heat treating at the temperatures specified in Table 3-2 are presented in Figure 3-2. The precipitation strengthened alloys such as Inconel 718 and Rene' 41 are characterized by precipitation of carbides. The fact that the Rene' 41 alloy shows a subsurface zone depleted of precipitates is attributed to the oxidation that occurred during the final heat treatment of the material. TDNiCr, a dispersion strengthened alloy, is characterized by very large directional grains and a fine dispersion of thoria. The recrystallized layer at the surface of the alloy was formed during the heat oxidation treatment as a result of the plastic deformation induced during roll-leveling the sheet. The columbium alloy FS-85 is shown with the intermetallic coating and diffusion zone. The columbium specimens were coated by Sylvania with



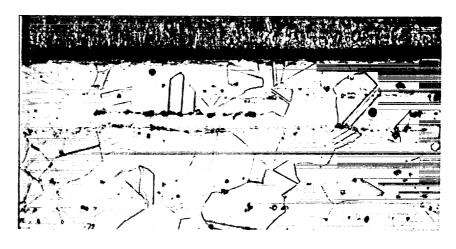
SCANNING ELECTRON MICROSCOPE PHOTOGRAPHS OF THE SURFACES OF HIGH TEMPERATURE ALLOYS AFTER FINAL HEAT TREATMENT

Figure 3-1

- .0762 mm (.003 inch) per side of fused slurry silicide, designated R512E. The coating process used was the same as that developed on the NASA program "Fused Slurry Silicide Coatings for Columbium Alloy Reentry Heat Shields," NAS-3-14307 (Reference 17). The thickness of the diffusion zone in Figure 3-2 was determined to be .025 mm (.001 inch). Because of their brittle nature, the contribution of the coating and the diffusion zone to the load bearing cross section can be assumed negligible. For this reason, subsequent strength calculations were based on the dimensions of each specimen prior to coating minus .025 mm (.001 inch) per side to compensate for the diffusion zone.
- 3.2 <u>Baseline Mechanical Properties</u> After the samples were given their final heat treatments, both smooth and fatigue precracked specimens were tensile tested to determine baseline properties. The results of these tests are presented in Table 3-3. All tensile tests are in the longitudinal direction (rolling direction). The longitudinal tests were selected to provide the type of data that would be representative of the tensile loading expected in the heat shield panels. The smooth tensile results presented in the table are above the minimum tensile values specified in the applicable materials specification and as a result these materials are representative of state-of-the-art technology for thin gage sheet. The data in Table 3-3 also indicate that strength reductions occur for most of the alloys tested in the fatigue precracked condition, with notched to unnotched strength ratios ranging from .67 to .97.
- 3.3 <u>Criteria for the Selection of Precracked Specimens</u> Stress corrosion susceptibility can be treated by classical fracture mechanics in terms of a critical stress intensity factor for the particular test environment. However, the use of thin gage sheet material precludes the measurement of valid fracture toughness parameters, especially at high temperatures where crack tip blunting occurs. The test specimens used in this program represented the conditions likely to be

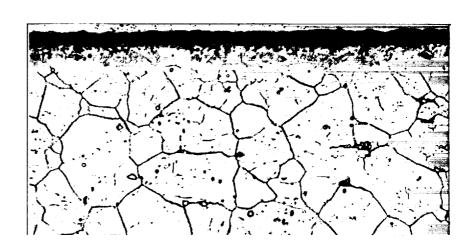
INCONEL 718 SOLUTION TREATED 1241⁰K-1/2 HR AGED 991⁰K - 8 HR + 894⁰K - 10 HR

> ETCHANT HCI + H₂O₂ MAG. 250X



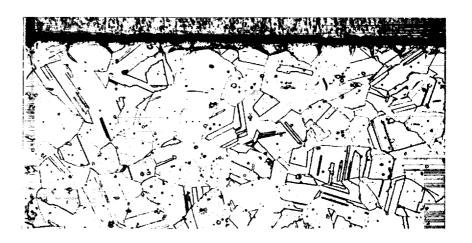
RENE'41 SOLUTION TREATED 1394⁰K 1/2 HR AGED 1172⁰K - 4 HR

> ETCHANT HCI + H₂O₂ MAG. 250X



L605 SOLUTION TREATED 1352°K

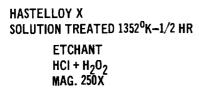
> ETCHANT HCI + H₂O₂ MAG. 250X

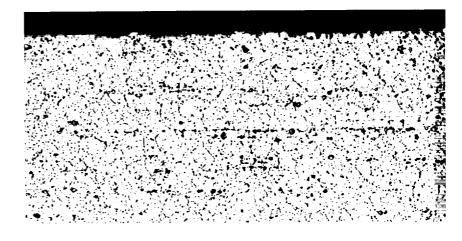


MICROSTRUCTURE OF HIGH TEMPERATURE MATERIALS AFTER HEAT TREATMENT

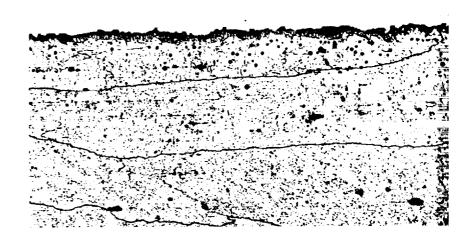
457-3035

Figure 3-2





TDNiCr SOLUTION TREATED 1449 $^{\rm O}$ K=1 HR ETCHANT HCI + ${\rm H_2O_2}$ MAG. 250X



FS-85
COATED WITH FUSED SLURRY
SILICIDE R-412E
1689⁰K-1 HR
ETCHANT
HNO₃
HF
LACTIC
MAG. 250X



MICROSTRUCTURE OF HIGH TEMPERATURE MATERIALS AFTER HEAT TREATMENT (Continued)

457-3037

Figure 3-2

ROOM TEMPERATURE TENSILE PROPERTIES FOR HIGH TEMPERATURE ALLOYS

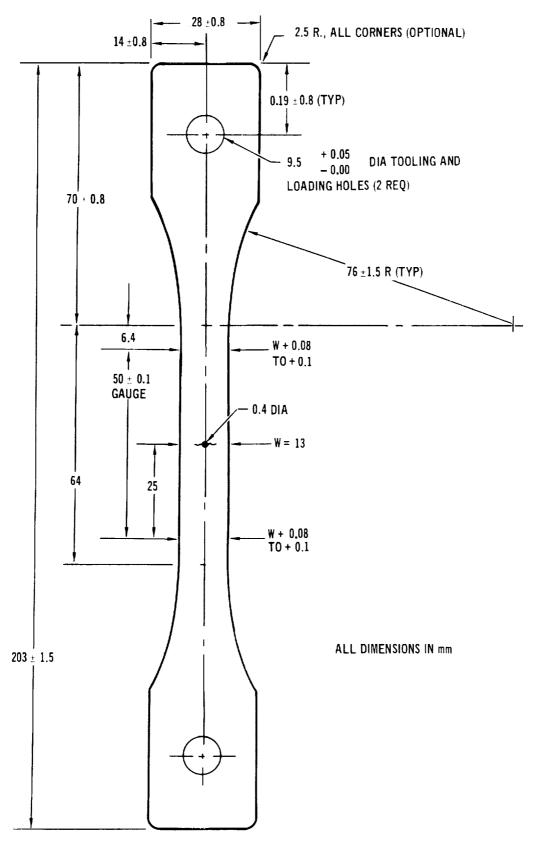
		UNNO	ТСНЕВ	JNNOTCHED PROPERTIES	TTES				NOTO	NOTCHED PROPERTIES*	ROPER :	ries*		
ALLOY	SPECIMEN	F _{ty} (0.2%)	1.2%)	F _{tu}		ELONGA- TION,	CDECIMEN	CRACK LENGTH	-ENGTH	Ftu	2	ELONGA-	F _{tu} (NOTCHED)	
		MN 'm²	KSI	MN/m ²	KSI	%,IN 5.08cm (2.0 IN.)		шш	<u>R</u>	MN/m ²	KSI	%,IN 5.08сm	F _{tu} (UNNOTCHED)	
INCONEL 718	A1	1150	167	1360	178	20								
	A2	1150	167	1370	199	20	A12	2.278	0.0897	1220	171	2.0		-
	A3	1160	<u></u> [22	1370	199	20	A42	2.869	0.1120	1320	192	1.5		
	AVG:	1150	167	1370	199	70				1270	184	1.8	0.92	
RENE'41	꿉	792	115	1090	158	10								
	R2	758	110	1070	155	14	R55	2.087	0.0822	814	118	0.5		
	22	977	113	1030	149	a	8	3.075	0.1211	869	126	0.5		
	AVG:	176	113	1060	154	11				841	122	0.5	0.79	
L-605		410	59.5	325	144	70								
	П	455	0.99	1010	146	55	L28	2.150	0.0846	662	96.0	3.5		
		451	65.5	1010	146	28		2.480	9/60.0	674	87.8	3.5		
	AVG:	439	63.7	981	145	61		•		699	96.9	3.4	0.67	
HASTELLOY X	×	334	48.4	792	115	37								
	X	345	50.0	813	118	24	X42	2.678	0.1055	585	84.8	3.0		
	\$	372	54.0	844	124	32	X12	2.744	0.1080	99	87.0	2.5		
	AVG:	350	50.8	816	119	31				265	85.9	2.8	0.72	
TDNIC	F	548	79.5	724	105	16								_
	21	551	80.0	737	107	16		2.875	0.1140	654	94.8	0.5		
	<u></u>	544	79.0	717	104	=	121	2.600	0.1024	635	92.1	0.5		-
	AVG:	547	79.5	726	105	14				644	93.4	0.5	0.89	
FS-85/	┎	474	8.8	619	89.9	* *								
R-512E**	F2	498	72.3	630	91.5	17	92	4.340	0.1709		84.9	3.5		
	Œ	485	70.4	618	89.7	17	FI7	1.540	9090'0	611	9.88	2.5		
	AVG:	486	70.5	622	90.3	17				602	87.2	3.0	0.97	
			1	1	1						1			_

**BASED ON SEM DETERMINATION OF CRACK LENGTH AFTER TEST
***BASED ON COLUMBIUM REMAINING AFTER COATING
****BROKE OUTSIDE GAGE

encountered for a metallic TPS even though the results are not expressed in fracture mechanics terminology.

Precracked tensile specimens were selected for use on this program primarily because such cracks are representative of the defects that can be introduced in sheet and structural components during normal manufacturing processes such as forming and welding. Such defects may escape detection during routine inspection procedures and subsequently lead to failure when the component is subject to proof-test or service loads. The existence of such prior defects has been demonstrated many times by analysis of delayed failures triggered by fatigue, stress-corrosion, and hydrogen embrittlement. Problems such as these have received much attention in the fabrication of rocket motor cases and pressure vessels which are subjected to high proof and operating stresses. Perhaps the most dramatic failure of this type occurred in a 660 cm (260 inch) diameter solid propellant booster which failed during proof test. The failure was attributed to buried weld defects which were not detected during routine inspection (Reference 16).

Precracked fracture toughness specimens came to be used in the study of such slow crack growth mechanisms as stress-corrosion. The use of such specimens led to the discovery (Reference 17) in 1964 that certain titanium alloys, previously considered immune to stress-corrosion in aqueous environments, failed catastrophically after short exposure times when fatigue cracked prior to loading. Since the susceptibility of a given material to stress-corrosion might be increased by the presence of a crack, fatigue precracked specimens were used for this program to ensure reliable results when evaluating stress-corrosion susceptibility. A further advantage was gained by using a precracked sample in that the excessively long incubation period required to form a crack in a smooth specimen was avoided without compromising the validity of the test results. The center-cracked-tension specimen shown in Figure 3-3 was selected because the thin gage to be studied



PRECRACKED TENSILE SPECIMEN

Figure 3-3

precluded a partial thickness crack, and it presented fewer problems in precracking than an edge-notch type.

3.4 <u>Precracking of Tensile Specimens</u> - The precracking was accomplished by drilling a .254 mm (.010 inch) to .508 mm (.020 inch) starter hole through the center of the gage length of the specimen and then subjecting each to tension-tension loading at a stress ratio of 0.10 on the Sonntag SF-1-U universal fatigue testing machine shown in Figure 3-4.



TENSILE SPECIMEN FATIGUE PRECRACKING APPARATUS

Figure 3-4

Initially, a maximum fatigue load, equivalent of 50% of the yield load was used for each alloy. This load was then increased until a load was found for each alloy that would initiate fatigue cracking within approximately 100,000 cycles.

After fatigue crack initiation in each specimen, the fatigue load was lowered to minimize plastic deformation at the tip of the fatigue crack. The fatigue crack growth was monitored with a strobe light and a stereoscope. The fatigue precracking was discontinued when the total crack length (crack length plus hole diameter) in each specimen reached approximately 2.54 mm (0.1 inch). A summary of the fatigue precracking is presented in Table 3-4.

Table 3-4

FATIGUE PRECRACKING DATA FOR NOTCHED TENSILE SPECIMENS

ALLOY	YIELD STRENGTH		NOMINAL STRE	GROSS SS (a)	PERCENTAGE OF	AVEPAGE Number of		OTAL K Length
7,001	MN/m ²	KSI	MN/m ²	KSI	YIELD STRENGTH	CYCLES	Cm	IN
INCONEL 718	1152.7	167.3	624.9 576.7	90.7 (b) 83.7 (c)	54.2 50.0	70,000 52,000	0.076 0.254	0.03 0.10
RENE'41	776.5	112.7	520.2 454.7	75.5 (b) 66.0 (c)	67.0 58.5	127.000 98,000	0.076 0.254	0.03 0.10
L-605	438.9	63.7	471.3 439.6	68.4 (b) 63.8 (c)	107.2 100.0	76,000 252,000	0.051 0.254	0.02 0.10
HASTELLOY X	350.0	50.8	403.1 322.5 248.7	58.5 (b) 46.8 (c) 36.1 (c)	115.0 92.3 71.0	107,000 167,000 233,000	0.051 0.152 0.254	0.02 0.06 0.10
TDNiCr	547.8	79.5	434.1 372.1	63.0 (b) 54.0 (c)	79.3 68.0	90,000 75,000	0.076 0.254	0.03 0.10
FS-85	485.7	70.5	237.0 207.4	34.4 (b) 30.1 (c)	48.1 42.8	70,000 60,000	0.076 0.254	0.03 0.10

⁽a) BASED ON ORIGINAL CROSS SECTION

457-3040

⁽b) STRESS REQUIRED TO FORM CRACK

⁽c) STRESS REQUIRED TO PROPAGATE CRACK

4.0 ALTERNATE IMMERSION EXPOSURE

4.1 Preparation of Test Specimens - Four precracked tensile specimens and two control coupons of each alloy were subjected to alternate immersion testing. The coupons, 25 x 51 mm (1 x 2 inches), were cleaned by rinsing in distilled water followed by rinsing in acetone and then were weighed to within ± 0.0001 gram. The tensile specimens were strain gaged .19 mm (0.75 inches) away from the crack. Two specimens of each alloy were then loaded to their final precracking load on the Baldwin Universal Testing Machine, and the total crack length in each specimen was measured to ± 0.0025 mm (0.0001 inches) with a cathetometer. A net section stress equal to 95% of the 0.2% yield strength listed in Table 3-3 was then applied to each specimen, and the strain for each specimen, as measured with a strain gage and a strain indicator, was recorded. After removal from the Universal Testing Machine, the specimens were placed in a constant strain corrosion test fixture and stressed in that fixture until the strain gages indicated that the strain corresponding to 95% of the 0.2% yield strength as determined in the Universal Testing Machine, was reached. The test fixtures were then masked with Unichrome 320 Stop-Off to prevent possible galvanic corrosion between the frames and specimens, and the strain gages and strain gage leads were masked with neoprene chem-mill maskant. The remaining two strain-gaged specimens of each alloy were held in reserve for further testing in case corrosion failures occurred in the specimen loaded to 95% of yield. A photograph of a precracked tensile specimen stressed in a constantstrain corrosion test fixture is presented as Figure 4-1.

4.2 Test Procedure - The stressed tensile specimens and the 25 x 51 mm (1 x 2 inch) control coupons were then subjected to alternate immersion in a 3.5% salt solution of simulated sea water, as specified in ASTM-D-114-52, Formula A. The sodium chloride content was checked daily with a salinometer, and regular adjustments

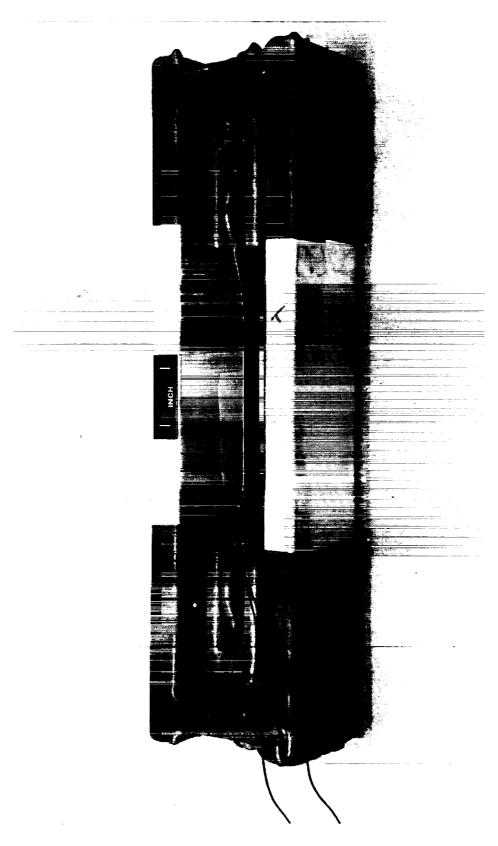


Figure 4-1

were made to maintain the weight percentage of the sea salt in the solution at 3.5 percent. The test cycle consisted of 10 minutes of saline immersion followed by 50 minutes of air exposure for a total of 60 days.

4.3 Results of Alternate Immersion Exposure of Unstressed Coupons - One coupon of each alloy was removed from testing, cleaned by rinsing in distilled water and acetone, weighed, and returned to this test after 15, 30 and 45 days of exposure. Both coupons of each alloy were removed from testing after 60 days exposure. The coupons were cleaned by rinsing in distilled water followed by rinsing in acetone. The specimens then were weighed and the weight change determined. The results of these measurements are presented in Table 4-1.

Photographs of the coupon surfaces after 60 days uninterrupted exposure were taken with the Scanning Electron Microscope and are shown in Figure 4-2. Comparison of these photographs with those in Figure 3-1 shows that, with the exception of Inconel 718, no change in surface appearance occurred as a result of alternate immersion testing. Although the surface of the Inconel 718 specimen shows a slight reaction, the photographs reveal no evidence of pitting or corrosion. The microstructures of these specimens, shown in Figure 4-3, confirm this conclusion, showing that the heat oxidized surface layer of all specimens is intact and the substrate unaffected.

Because a sufficient quantity of L-605 was available, control specimens machined from as-received material were included in this phase of the program in order to ascertain the effect of a surface oxide layer on corrosion behavior. The data in Table 4-1 indicate that L-605 is slightly more susceptible to corrosion in the heat oxidized condition than in the as-received condition. However, as shown in Figures 4-2 and 4-3, the heat oxidized L-605 specimen did not exhibit any pitting as a result of the exposure.

Fable 4−1

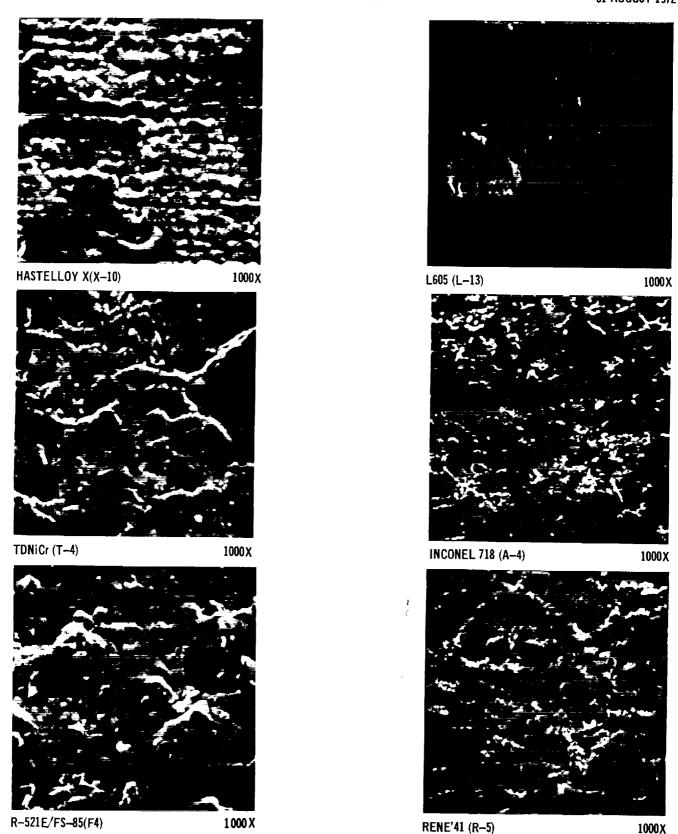
WEIGHT CHANGE OF COUPONS EXPOSED TO ALTERNATE IMMERSION STRESS CORROSION TEST

ALLOY	SPECIMEN #	WT. BEFORE WT. AFTER EXPOSURE 15 DAYS EXP (GRAMS)	VT. BEFORE WT. AFTER EXPOSURE 15 DAYS EXP (GRAMS)	WT. CHANGE AFTER 15 DAYS (MG)		WT.CHANGE AFTER 30 DAYS(MG)	ď,	WT. CHANGE AFTER 45 DAYS (MG)	WT. AFTER 60 DAYS EXP. (GRAMS)	NET WEIGHT CHANGE (MG)
RENE'41	R95	2,7616	2.7612	-0.4	2.7611	-0.5	2.7611	-0.5	2.7613	-0.3
	R104	2,7236	ı	1	I	ı	ı	ī	2,7234	-0.2
INCONEL 718	A77	4,5323	4.5327	+0.4	4.5327	+0.4	4,5323	0	4.5330	+0.7
-	A79	4.5040	ı	ř	ŀ	i	1	1	4.5050	+1.0
TDNiCr	T62	3,4220	3.4217	-0.3	3.4215	-0.5	3.4219	-0.1	3, 4220	0
	T89	3,4869	1	ı	1	I	ı	ı	3.4871	+0.2
L605 (1)	۲۷8	3,1670	3, 1650	-2.0	3.1647	-2.3	3.1645	-2.5	3.1645	-2.5
	L97	3,2439	1	ı	ı	1	ı	ı	3.2423	-1.6
L605 (2)	L95	3,3501	3,3500	-0.1	3.3502	+0.1	3,3502	- -	3,3506	+0.5
	L100	3,2739	I	ŧ	ı	I	ı	ı	3.2735	-0.4
HAYNES 188	H63	3,1199	3.1200	+0.1	3,1199	0	3.1201	+0.2	3.1202	+0.3
	16Н	3.0780	ı	ı	ı	1	ı	i	3.0787	+0.7
HASTELLOY X		2,6891	2.6887	-0.4	2.6889	-0.2	2.6889	-0.2	2.6890	-0.1
	X102	2,6945	Ī	ı	I	ı	1	ı	2.6943	-0.2
FS-85/R512E	F83	5.0212	5.0219	+0.7	5.0269	+5.7	5.0228	+1.6	5.0243	+ 3.1(3)
	F84	5.0158	5.0164	+0.6	5.0215	+5.7	5.0179	+2.1	5,0200	+ 4.2(3)

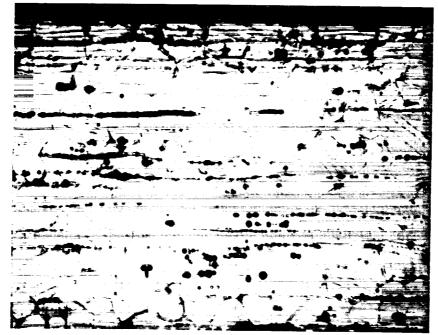
(1) THIS SPECIMEN WAS HEAT OXIDIZED (1950°F TO 1975°F FOR 30 MINUTES IN AIR)

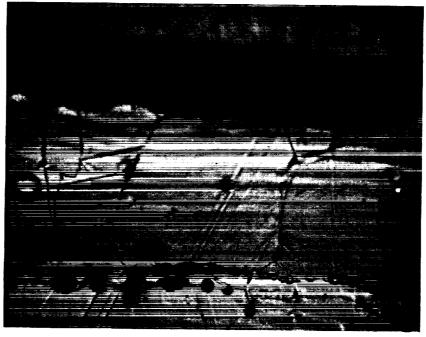
THIS SPECIMEN WAS TESTED IN THE "AS RECEIVED" CONDITION

(3) WHEN THESE SPECIMENS WERE LATER ULTRASONICALLY CLEANED, THE "WEIGHT CHANGE" BECAME -1.2 AND -3.0 MG, RESPECTIVELY.



SURFACE OF ALTERNATE IMMERSION SPECIMENS AFTER 60 DAYS OF EXPOSURE

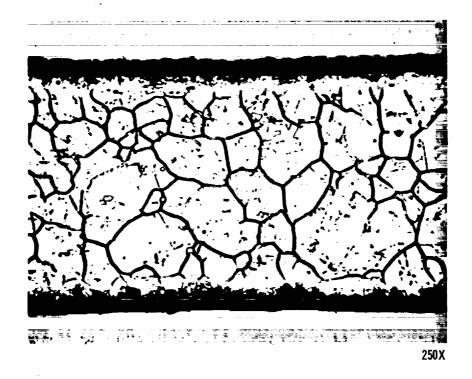




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MICROSTRUCTURE OF INCONEL 718 UNSTRESSED CONTROL COUPON A-79 AFTER 60 DAYS ALTERNATE SALT WATER IMMERSION

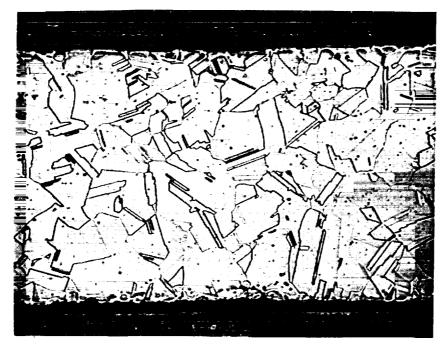
Figure 4-3 (a)

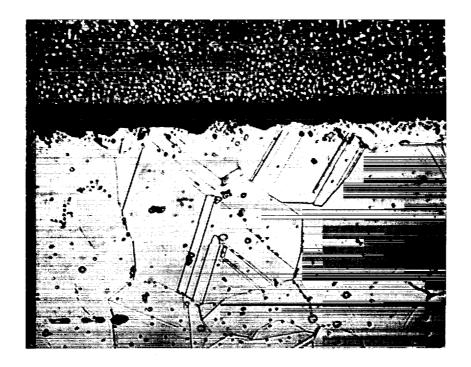




MICROSTRUCTURE OF RENE'41 UNSTRESSED CONTROL COUPON R-104 AFTER 60 DAYS ALTERNATE SALT WATER IMMERSION

Figure 4-3 (b)

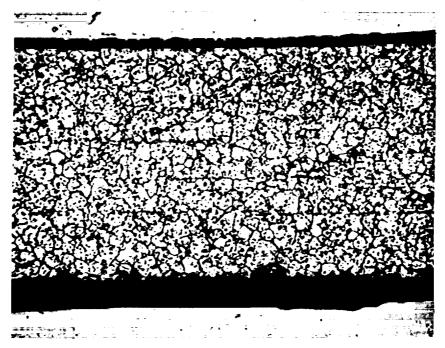


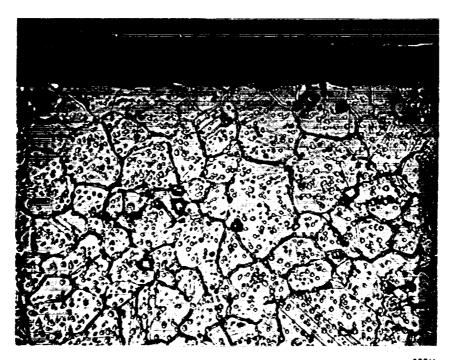


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MICROSTRUCTURE OF L-605 UNSTRESSED CONTROL COUPON L-97
AFTER 60 DAYS ALTERNATE SALT WATER IMMERSION

Figure 4-3 (c)



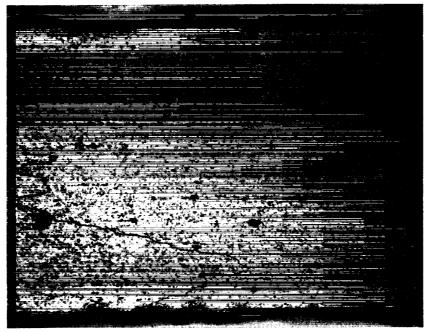


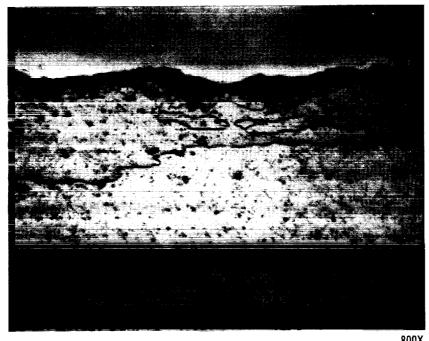
800X

MICROSTRUCTURE OF HASTERLLOY X UNSTRESSED CONTROL SPECIMEN X-102 AFTER 60 DAYS ALTERNATE SALT WATER IMMERSION

457--3047

Figure 4-3 (d)

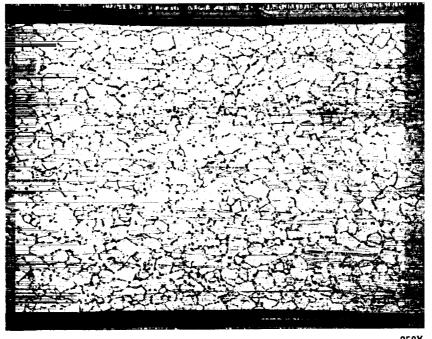


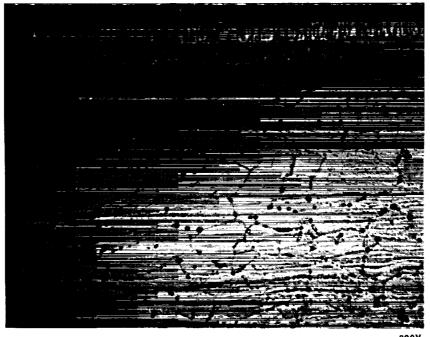


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MICROSTRUCTURE OF TDNIC UNSTRESSED CONTROL SPECIMEN T-89 AFTER **60 DAYS ALTERNATE SALT WATER IMMERSION**

Figure 4-3 (e)





800X

MICROSTRUCTURE OF FS-85 UNSTRESSED CONTROL SPECIMEN F-84 AFTER 60 DAYS ALTERNATE SALT WATER IMMERSION

457 - 3049

Figure 4-3 (f)

Since the $\bar{L}-60\bar{5}$ was the only cobalt based alloy in the test and did exhibit high weight losses, it was decided to test Haynes 188 in alternate immersion to see if the weight loss is a function of the cobalt percentage. Haynes 188, which contains 12 weight percent more nickel than L-605, is essentially L-605 with lanthanum added to stabilize the cobalt oxide above. From Table 4-1 it can be seen that the change in weight of Haynes 188 is more consistent with the other superalloys. However, even with the larger weight loss of the L-605 specimens there does not appear to be any evidence of corrosion in Figure 4-2.

The data in Table 4-1 also shows that the FS-85 specimens had the largest weight gain for any alloy investigated. Since these specimens exhibited a consistent weight gain after each measurement, it was suspected that salt and dirt had become entrapped in the rough coating surface. The specimens were ultrasonically cleaned after 60 days exposure, and the total weight changes became -1.2 and -3.0 mg. Although the weight change data is erratic, the photomicrograph in Figure 4-2 again shows no evidence of pitting or general corrosion.

4.4 Results of Alternate Immersion Exposure of Stressed Tensile Specimens Because no corrosion failures of the stressed tensile specimens occurred after
60 days of alternate immersion exposure, the specimens were removed from the test
fixtures and subjected to tensile tests to determine their residual ultimate
strength. The tensile test data is presented in Table 4-2, along with the baseline
notched tensile data from Table 3-3. From this data it is apparent that there
were no strength reductions in any of the alloys tested.

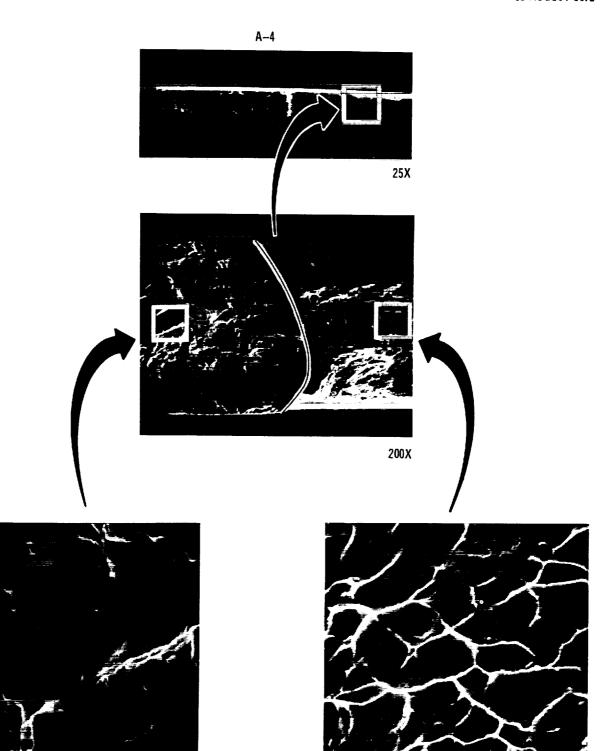
The fracture surfaces of the precracked tensile specimens were examined under the Scanning Electron Microscope (SEM) for evidence of crack propagation. Based on the results of this examination there does not appear to be any evidence of crack growth as a result of the 60-day exposure; all fracture surfaces were ductile.

Table 4-2
EFFECT OF ALTERNATE IMMERSION ON THE ROOM TEMPERATURE NOTCHED
TENSILE STRENGTH OF HIGH TEMPERATURE ALLOYS

ALLOY	•••	EFORE E IMMERSION	F _{tn} AFTER ALTERNATE IMMERSION			
i	MN/m ²	KSI	MN/m ²	KSI		
INCONEL 718	1220 1320	177 1 9 2	1300 1310	189 190		
AVG:	1270	184	1300	190		
RENE'41	814 869	118 126	859 885	125 128		
AVG:	841	122	872	126		
L-605	662 674	96.0 97.8	621 662	90.0 96.1		
AVG:	669	96.9	640	93.0		
HASTELLOY X	585 600	84.8 87.0	596 597	86.5 86.6		
AVG:	592	85.9	596	86.6		
TDNiCr	654 635	94.8 92.1	661 666	95.8 96.6		
AVG:	644	93.4	664	96.2		
FS-85/R-512E	592 611	85.9 88.6	609 633	88.4 91.9		
AVG:	602	87.2	621	90,2		

A typical photograph of a fracture surface of an Inconel 718 specimen is shown in Figure 4-4. The fracture surfaces of the other specimens show the same transition from fatigue precracking to tensile failure without any indication of brittle failure that would be associated with stress corrosion susceptibility.

All of the tensile failure zones are of the dimple rupture type which characterizes a ductile material. During the fractographic examination of the specimens it was determined that the original crack length was longer than anticipated and, as a result, most of the precracked specimens were subjected to a net section tensile stress greater than 95% of the 0.2% yield strength. However, in no case were any of the specimens stressed more than 90% of their notched tensile strength. The actual net section stresses to which the specimens were subjected are presented in Table 4-3.



FRACTURE SURFACE OF INCONEL 718 SPECIMEN AFTER 60 DAY ALTERNATE IMMERSION TEST

3000X

457-3051 Figure 4-4

ACTUAL APPLIED STRESSES FOR ALTERNATE IMMERSION TESTING OF HIGH TEMPERATURE ALLOYS

	BASELINE PROPERTIES	ERTIES		APPLIED \$	APPLIED STRESS FOR ALTERNATE IMMERSION TESTING	RNATE IMMERSIO	N TESTING	
Ftn (NOTCHED SPECIMEN)	E	JIMEN)	BASED ON	BASED ON 95% OF 0.2% F _{ty}	BASED ON SEM DETERMINATION OF CRACK LENGTH	ETERMINATION LENGTH	ACTUAL PERCENT	PERCENT OF F.
MN/m²		ksi	MN/m ²	ksi	MN/m ²	ksi	Ur F _{ty}	5
1270		184	1100	160	1090 1110	158 161	95 96	86
841		122	738	107	750 761	109 110	97 98	88 06
699		6:96	417	60.5	413 422	59.9 61.2	96 96	88
265	1	85.9	333	48.3	332 337	48.2 48.9	95 96	56 57
644		93.4	521	75.5	561 548	81.4 79.4	101 100	87 85
709	į.	87.2	462	67.0	495 51 4	71.8 74.6	101	85

*BASED ON COLUMBIUM REMAINING AFTER COATING

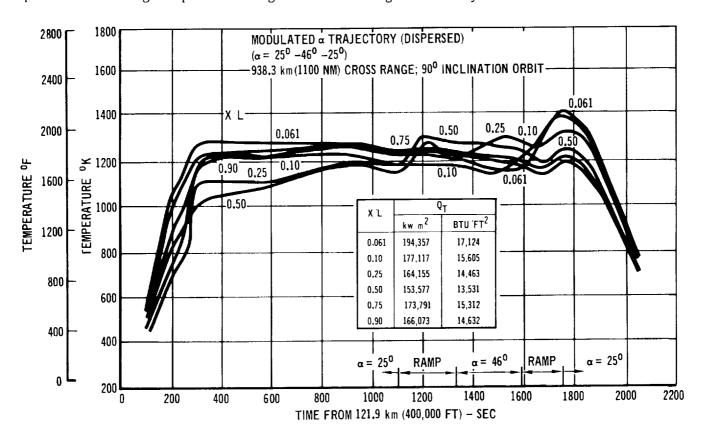
4.5 Conclusions - Since there were no tensile failures in any of the alloys during the 60-day alternate immersion test and no evidence of pitting or general corrosion during the same exposure period, it is concluded that these materials are immune to corrosion or stress corrosion under this set of test conditions.

Figure 5-1

5.0 ELEVATED TEMPERATURE TESTING

<u>5.1 Selection of Test Conditions</u> - The purpose of this part of the program was to determine the effect of salt coatings on the high temperatures mechanical properties of metallic TPS materials subjected to reentry conditions.

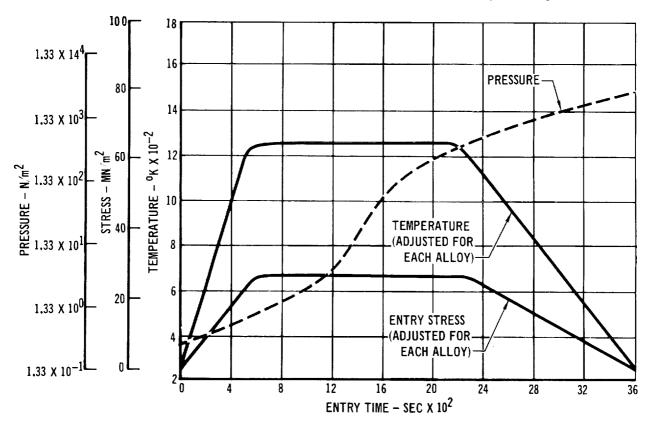
To accomplish this task an analysis was made of the delta orbiter trajectory taken from the McDonnell-Douglas Space Shuttle Phase B final report. The time-temperature history, presented in Figure 5-1, was determined by assuming that all local aerodynamic heating is rejected from the vehicle outer moldline by thermal radiation at each instant during entry. All temperatures were obtained considering an 0.8 surface emittance radiating to a cold sink. All of the time-temperature curves consist of an initial heat-up followed by an interval of modulating heating prior to heating drop-off during the later stages of entry.



DELTA ORBITER - BOTTOM CENTERLINE TEMPERATURES

DELTA ORBITER - BOTTOM CENTERLINE TEMPERATORES

Since the trajectory evaluation will continue throughout the various phases of the Shuttle development, it was deemed appropriate to generate a simplified time-temperature curve which was not connected to any specific trajectory but emcompassed the most severe elements of any probable trajectory. The simplified curve was required to retain as much resemblance as possible to actual entry trajectories. With this objective in mind, a resultant time-temperature curve was developed. This curve, which is shown in Figure 5-2, is very close to the thermal profile of the Phase B Delta Orbiter (Figure 5-1) with respect to maximum time at temperature. The times required to reach maximum temperature and to cool down are different but this is due to the times required for furnace heat-up and cool down rather than to any specific change in trajectory rationale. During the actual testing of the high temperature materials the maximum temperature was adjusted to correspond to the maximum use temperature of the specific alloys being studied.



ENTRY TEST CYCLE

Figure 5-2

CORROSION OF

HIGH TEMP ALLOYS

The pressure curve seen in Figure 5-2 represents the expected TPS surface pressure as a function of entry time. The pressure curve was obtained from the expected altitude versus time plot of the Phase B Delta Orbiter shown in Figure 5-3. From this figure the altitude plot was converted to an ambient-at-altitude pressure for the test profile. The ambient-at-altitude pressure represents the pressure on the inside of the panels where entrapped salt is likely to accumulate. The salt deposits on the outside of the vehicle would probably be washed into crevices or inside the panels during rain or heavy dew. As a result, the test pressure should be representative of the inside of the panel rather than the outside.

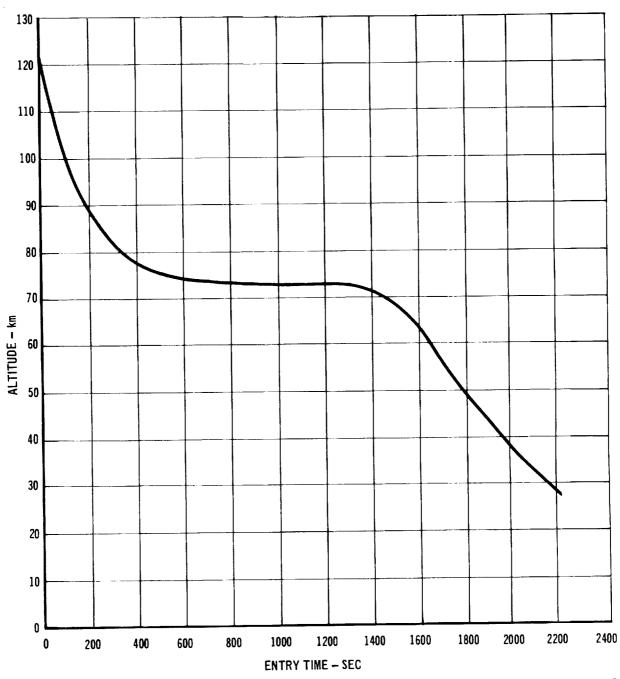
In addition to temperature and pressure, the TPS will be subjected to an applied stress due to pressure differentials across the TPS panels. The stress within the panel can be either tension or compression depending upon the point within the corrugation being examined. Since stresses within a panel are complex and difficult to separate into pure tensile elements, it was decided to select a tensile stress based on creep limits for the testing of high temperature materials. The creep limit selected was 0.5% after 50 flights. Any plastic deformation over this amount would cause excessive plastic deformation within the panel exposing it to excessive aerodynamic heating. The stress profile is presented in Figure 5-2 and the maximum stress level at maximum temperature is presented in Table 5-1. The stresses shown in Table 5-1 are net section cyclic stresses required to produce less than 0.5% plastic deformation after 50 entry flights. These stresses are less than those obtained from a constant stress, constant temperature creep curve. The lower stresses were selected because in-house tests conducted at McDonnell Douglas (Reference 18) have shown that creep strains greater than those predicted from constant stress-constant temperature data can be obtained by cyclic

CORROSION OF

457-3055

HIGH TEMP ALLOYS

stress and temperature conditions similar to shuttle flight conditions. Therefore, the lower stresses are based on previously developed in-house data.



ENTRY TRAJECTORY FOR DELTA ORBITER

Figure 5-3

5.2 Salt Thickness Determination - As discussed in Section 2.2, the amount of salt deposited on a specimen has a direct bearing on the stress corrosion susceptibil-

Table 5-1
TEST CONDITIONS FOR ELEVATED TEMPERATURE TESTING

	PEA TEST TEMP		PE Applied		
ALLOY	οK	°F	MN/m ²	ksi	SPECIMEN CONDITION
INCONEL 718	811	1000	220	32	SALT COATED
			220	32	UNCOATED
	922	1200	165	24	SALT COATED
			165	24	UNCOATED
RENE '41	1033	1400	117	17	SALT COATED
			117	17	UNCOATED
	1144	1600	58.6	8.5	SALT COATED
			58.6	8.5	UNCOATED
L605	1144	1600	34.5	5.0	SALT COATED
			34.5	5.0	UNCOATED
	1255	1800	13.8	2.0	SALT COATED
			13.8	2.0	UNCOATED
HASTELLOY X	1255	1800	6.9	1.0	SALT COATED
			6.9	1.0	UNCOATED
	1366	2000	3.5	0.5	SALT COATED
			3.5	0.5	UNCOATED
TDNiCr	1366	2000	34.5	5.0	SALT COATED
			34.5	5.0	UNCOATED
	1477	2200	24.1	3.5	SALT COATED
(1)			24.1	3.5	UNCOATED
R512E/FS-85	1477	2200	68.9	10	SALT COATED
			68.9	10	UNCOATED
	1589	2400	44.8	6.5	SALT COATED
			44.8	6.5	UNCOATED

457-3056 (1)BASED ON COLUMBIUM REMAINING AFTER COATING.

ity of some alloys. In order to insure that the elevated temperature testing of salt precoated specimens simulates the conditions the shuttle is expected to encounter in service, the nominal thickness of salt that would be deposited on the TPS at Cape Kennedy had to be determined.

In Reference (19) it was found that the average concentration of salt in the air at the immediate coastline is 9 times that of the concentration that is present in an area several miles inland from the coast. To support this statement, the difference in the salt particle content in air at Cape Kennedy and Washington, D. C. was cited.

Location

Salt Particle Concentration in Air

Cape Kennedy

$$7.2 \times 10^{-6} \text{ g/m}^3$$

Washington, D. C.

$$0.8 \times 10^{-6} \text{ g/m}^3$$

Since all of the salt suspended in air does not settle out, the settling rate had to be determined. Reference (20) stated that the amount of salt that settled on a surface at a point 12.1 km (7.5 miles) inland from an estuary was 1 milligram/m² - day. Based on this data it was assumed that the salt settling rate at Washington, D. C. (which is approximately 19.3 km (12 miles) from salt water) would be 1 mg/m² - day. The rate of deposition, r_D , can then be determined as follows:

$$r_D = 365 \text{ days/year} \times 1 \times 10^{-3} \text{ g/m}^2 - \text{day}$$

= .365 g/m² - year.

By assuming that the rate of settling is proportional to the quantity of salt suspended in the air, the amount of salt settling at Cape Kennedy (K) would be:

$$K = \frac{.365g}{^{2}_{m}-year} \times \frac{7.2 \times 10^{-6} g/m^{2} \text{ (Cape Kennedy)}}{0.8 \times 10^{-6} g/m^{2} \text{ (Washington D.C.)}}$$

$$= 3.285 \text{ g/m}^2 - \text{year}$$

The thickness of this deposit is determined by simple dividing the rate of deposit by the density of the salt (2.165 g/cm^3).

$$t = \frac{3.295g}{m^2 - year} \times \frac{1 \text{ cm}^3}{2.165g} \times \frac{m^2}{10^4 \text{ cm}^2} \times \frac{10 \text{ mm}}{1 \text{ cm}}$$

$$t = 1.52 \times 10^{-3} \text{ mm/year} (5.98 \times 10^{-5} \text{ in/year})$$

Allowing a safety factor of 10 to cover all possible errors in assumptions and differences in locations, the salt layer deposited on a surface is:

t = 0.015 mm/year (.0006 inch/year)

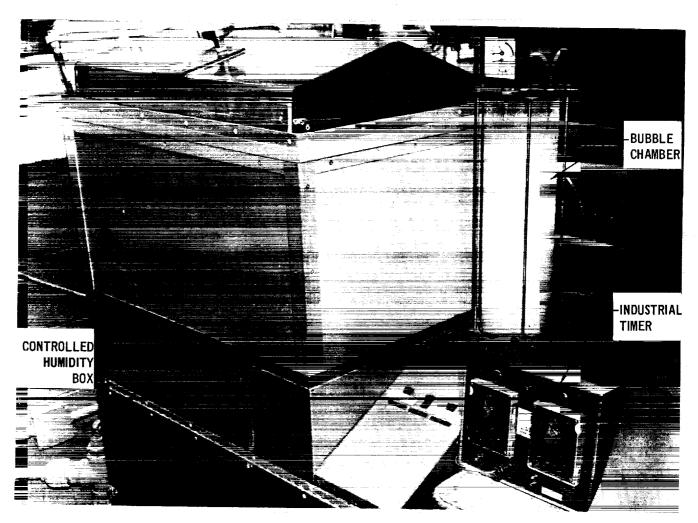
Based on the preceeding calculations, a salt coating of approximately 0.013 mm (.0005 inch) to 2.025 mm (.001 inch) was deemed adequate for elevated temperature testing purposes.

5.3 Application of Uniform Salt Coatings - The studies referenced in Section 2.2 have shown that, for some metals, corrosion susceptibility is directly proportional to the thickness of the salt deposit. A technique, then, had to be devised to produce a uniform and reproducible salt coating so as to make the test conditions representative of those the shuttle would see in actual service.

Several methods were investigated to produce a uniform salt coating of .013 mm to .025 mm on the test specimens. Pouring a salt solution over the specimens and allowing the water to evaporate produces a nonuniform coating with salt crystal agglomerates and thicknesses up to .10 mm (.004 inch). Spraying the salt onto the specimen with an air brush was deemed unsatisfactory because the salt film produced by this technique was irregular in thickness and nonadherent. The approach that give the best results was to allow the salt to settle from the air onto the surface as it would in actual service life.

A special salt settling chamber was required for applying the salt in this manner. This chamber, shown in Figure 5-4, consists of a controlled humidity box, a bubble chamber, and an industrial timer. The inside of the settling chamber is shown in Figure 5-5(a). Salt precoating is accomplished by injecting a fine spray composed of 7% synthetic sea water into the chamber, which is maintained at a constant temperature of 322°K (120°F). The spray strikes a splatter plate which divides it into a fine mist; any water droplets that are left on the splatter plate are siphoned off so that they do not affect the humidity in the chamber. In the actual operation of the equipment, the tensile specimens, coupons, and a clean glass control plate are placed in the chamber as shown in Figure 5-5(b). The

chamber is heated to 322°K (120°F) and the salt water is sprayed into the chamber for 15 minutes; after spraying, the salt is allowed to settle. At the completion of the 50-minute cycle, the spray cycle is repeated. After 30 cycles, the control plate is removed for measurement, the specimens turned over, and the process repeated to coat the other side. This technique produced a uniform, adherent coating as can be seen in the Scanning Electron Microscope photographs of the glass control plate in Figure 5-6.

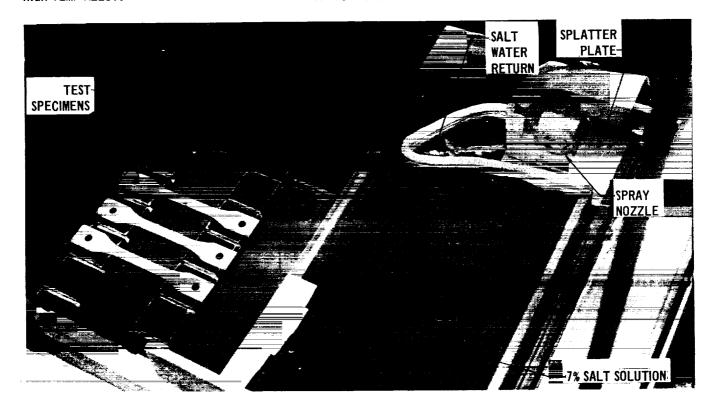


457-3057

SALT SETTLING CHAMBER

Figure 5-4

The average thickness of the salt deposit on the glass control plates after 30 cycles was found to be .013 mm (.0005 inch). The measurements were made using a depth microscope equipped with a dial indicator that had an



INTERIOR VIEW OF SALT SETTLING CHAMBER

FIGURE 5-5 (a)



457-3059

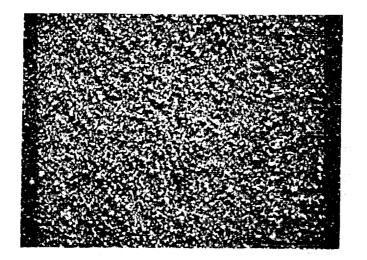
IN POSITION PRIOR TO SALT COATING

FIGURE 5-5 (b)

CORROSION OF HIGH TEMP ALLOYS

Yearly Summary Report NAS8-27270

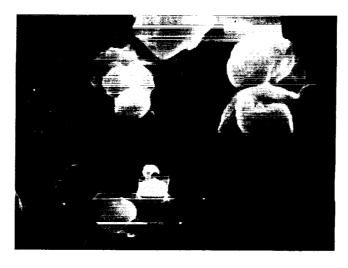
MDC E0609 31 AUGUST 1972



100 X



1,000 X



10,000 X

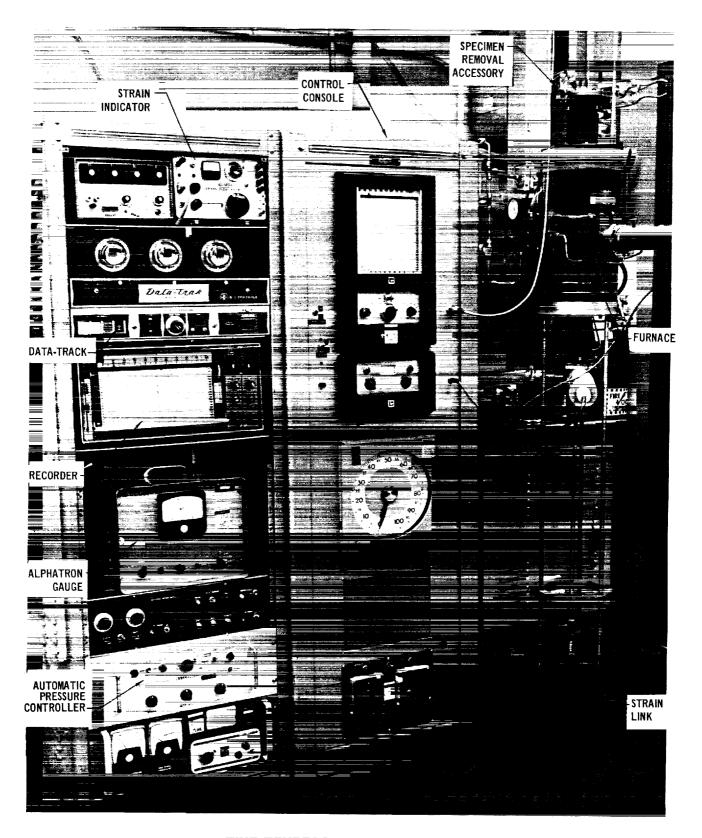
SCANNING ELECTRON MICROSCOPE EXAMINATION OF SALT PRECOAT PRODUCED BY THE SALT SETTLING CHAMBER

1.153

focusing on the control plate at high magnification and then refocusing on the top of the salt crystals, the difference in the readings on the dial indicator being the thickness of the salt deposit.

5.4 Exposure of Precracked Specimens - After the elevated temperature test profile, shown in Figure 5-2, had been established it was programmed into the reentry simulator shown in Figure 5-7. This simulator is basically a modified 63.5 mm (2-1/2 inches) diameter tube furnace manufactured by Astro-Industries with the necessary support equipment to automatically and simultaneously vary temperature, pressure, and stress with time. The furnace itself has an operating range of 533° to 2255°K (500° to 3600°F) at flowing air pressures of 1.01 x 10⁵ to 1.33 x 10⁻¹ N/m² (760 to 10⁻³ torr). The furnace was modified by removing the top and bottom hearths and installing in their place a muffle tube "0" ring sealed at both ends by means of a water-cooled housing and a spacer plate. The water-cooled upper closure contains a movable specimen holder and can be removed for specimen loading and unloading. The water-cooled lower closure contains provisions for connecting the vacuum detector, the pumping system, and the loading mechanism.

The equipment required for automatic pressure control includes an Alphatron vacuum gage and a pressure controller. The pressure is measured by a transducer in the Alphatron gage and converted into electric signals. These signals are monitored by the pressure controller, which compares them to the signals previously programmed on the Data-Trak. If there is a variation, the transducer will trigger a servovalve which will open or close as required to maintain the programmed pressure. An airflow rate of $16.4 \text{ cm}^3/\text{sec}$ (60 in 3/min) can be obtained at a pressure of $1.33 \times 10^3 \text{ N/m}^2$ (10 torr).



TIME-TEMPERATURE-PRESSURE-STRESS FACILITY

Figure 5-7

The programmed load is controlled by a 20 rpm reversible motor. A strain link between the motor and the tie rod is programmed into a strain indicator. The strain indicator compares the applied load with the programmed load on the Data-Trak and controls the motor accordingly. The temperature is monitored by a continuous reading optical pyrometer and is controlled by a platinum thermocouple, which is connected to a recorder and the Data-Trak.

After the furnace was programmed, four precracked specimens of each alloy were subjected to the simulated reentry environment shown in Figure 5-2. Of the four precracked specimens, two salt precoated and two uncoated specimens were tested to the conditions shown in Table 5-1. For the salt-coated specimens, the area around the crack was salt coated using the technique described in Section 5.3. Periodic inspections were made of the specimens after 5, 10, 15, 25, and 50 entry cycles. The inspections consisted of surface examination for evidence of pitting, creep measurements, and crack length measurements. During the visual examinations, if cracks were not coated with salt, the specimens were recoated. In addition to cycling tensile specimens, control samples were included in each furnace run to separate the effects of stress and precracking on the corrosion resistance of these materials from the effects of temperature and pressure. The controls were run in the salted and unsalted condition and then examined after 50 flights for weight change and evidence of pitting.

5.5 Results of Elevated Temperature Testing - Before the specimens were subjected to elevated temperatures, the gage length on every tensile specimen was marked off with either high temperature paint or scribe marks. In the case of the nickel and cobalt base superalloys, two dots of high temperature paint, Pyromark High Emittance Black, were placed 25.4 mm (1 inch) apart in the center of the gage length of each sample. The distance between these marks was determined to an

accuracy of ±0.025 mm (0.001 inch) using a Unitron measuring microscope. This microscope is equipped with a lense grid and a calibrated stage. Measurements were made by lining up a fixed location of one of the dots under the grid lines of the microscope, the calibrated stage was then moved until the grid lined up to a similar point on the other Pyromark dot. The distance the stage traversed is the gage length. The high emittance paint was selected because previous experience showed that scribe marks tended to be obscured by the materials oxide, making subsequent measurement difficult, if not impossible. Scribe marks were used on the coated columbium since these marks are not obscured by oxidation.

The gage lengths were remeasured after every 5, 10, 15, 25, and 50 entry cycles. The measurements were then converted into the percent creep, as shown in Table 5-2. From this data it can be seen that with the exception of two uncoated specimens of Hastelloy X and one salt coated specimen of Rene' 41, all of the creep deformations were below the one percent creep limit criterion. The creep deformations for these specimens were 1.22, 1.09 and 1.04 percent, respectively. It is to be noted, however, that because of the difficulty in the determining changes in crack length and specimen elongation, the data in Table 5-2 should be used for comparative purposes only. Because several specimens had to be recoated with salt, it was necessary to apply new reference marks since the new salt coating tended to obscure the old marks. Furthermore, the applied stresses on any specimen may have been greater than anticipated, since adjustments were made in the applied load to compensate for changes in cross section as crack growth occurred. It was difficult to get an exact determination of the crack length because in many instances it was camouflaged by oxides, as discussed in greater detail below. As a result, some of the applied loads may have been greater than anticipated, causing higher creep rates.

Table 5-2
RESULTS OF CREEP MEASUREMENTS AFTER ENTRY SIMULATION

	TF	MP	STR	22	SPECIMEN			% CREEP	AFTER CYCL	.ES ⁽¹⁾	
ALLOY	٥K	0F	MN/m ²		NUMBER	CONDITION	5	10	15	25	50
INCONEL 718	811	1000	220	32	A10	SALT COATED	0.24	0.29	0.42	0.47	0.56
					A17	SALT COATED	0,05	0,12	0.20	0.30	0.42
					A29	UNCOATED	0.09	0.18	0.27	0.34	0.45
					A 7	UNCOATED	0,13	0.16	0.25	0.35	0.47
	922	1200	165	24	A19	SALT COATED	0.04	0.12	0,23	0.39	0.48
					A28	SALT COATED	0.06	0.12	0.21	0.28	0.39
					A50	UNCOATED	0.08	80.0	0.14	0.26	0.49
					A52	UNCOATED	0.10	0.17	0.22	0.34	0.44
						CALT COATED	0.55	0.78	0.83	0.95	1.04
RENE'41	1033	1400	117	17	R9	SALT COATED SALT COATED	0.01	0.78	0.23	0.27	0.36
					R10	UNCOATED	0.47	0.58	0.62	0.64	0.73
į				l	R13		0.03	0.05	0.13	0.16	0.75
				١	R15	UNCOATED					
	1144	1600	58,6	8.5	R11	SALT COATED	0.10	80.0	0.10	NM	0.13
			1		R12	SALT COATED	0.08	-0.09	- 0.06	0.01	0.04
	١,				R16	UNCOATED	0.06	0.13	0.07	0,05	0.04
				l	R17	UNCOATED	0.26	0.11	0.12	0.17	0.42
L605	,,,,	1600	34.5	5.0	L15	SALT COATED	0.07	0.06	0.04	0.10	0.11
	1144	1000	34.5	3.0	L15	SALT COATED	0.02	NM	0.11	0.02	0.18
				l	L51	UNCOATED	0.14	0.15	0.18	0.17	0,19
					L53	UNCOATED	0.14	0.15	0.11	0.11	0.43
											SF ⁽²⁾
	1255	1800	13.8	2.0	L47	SALT COATED	NM	NM	0.10	0.14	0.40
				1	L50	SALT COATED	NM	NM	0.00	0.05	
			l	ŀ	L16	SALT COATED	0.08	0.28	0,46	0.61	0.74(3)
	'			ì	L56	UNCOATED	0.15	0.33	0.47	0.64	0.71
				ŀ	L57	UNCOATED	0.04	0.33	0.34	0.42	0.42
HASTELLOV X	1255	1800	6.9	1.0	X21	SALT COATED	0.09	-0.13	-0.02	0.15	0.20
HASTELLOY X					X27	SALT COATED	0.02	0.13	0.18	0.32	0.49
				ļ	X15	UNCOATED	0.21	0.27	0.46	0.61	0.75
	1			l	X36	UNCOATED	0.46	0.47	0.47	0.57	0.65
			1 25	٦٠	1	SALT COATED	0.01	0.39	0.45	0.45	0.50
	1366	2000	3.5	0.5	X28	SALT COATED	0.01	0.27	0.27	0.33	0.48
					X35	1	0.22	1.13	0.83	1.13	1,22
	ĺ			ļ	X39 X41	UNCOATED	0.63	0.63	0.64	0.92	1.09
				1	V41	UNCOATED			1		
TDNiCr	1366	2000	34:5	5.0	T22	SALT COATED	NM	0.02	0.04	0.32	0.43
				1	T23	SALT COATED	0.23	0.27	0.30	0.30	0.30
					T20	UNCOATED	0.23	0.67	0.83	0.86	0.89 0.13
			1	l	T35	UNCOATED	0.02	0.12	0.12	0.12	0,13
	1477	2200	24.1	3.5	17	SALT COATED	0.28	0.47	0.47	0.73	0,81
] **''	2200	67.1	13.3	T25	SALT COATED	0.17	0,16	0.26	0.19	0.26
	1	l		1	T51	UNCOATED	0.26	0.36	0.40	0.42	0.45
		1	1		T53	UNCOATED	0.42	0.54	0.62	0.66	0.78
				1			0.22	0,29	0.36	0.50	0.61
FS85/R512E	1477	2200	68.9	10	F8	SALT COATED	0.22	0.29	0.23	0.30	0.44
	1			1	F9	SALT COATED		I	0.23	0.20	0.33
				1	F13	UNCOATED	0.03 0.08	0.06	0.13	0.20	0.43
	1			1	F14	UNCOATED	0.00	0.10			
	1589	2400	44,8	6.5	F11	SALT COATED	0.42	0.59	0.69	0,78	0.82
		- 100		1""	F12	SALT COATED	0.29	0.36	0.43	0.54	0.68
			1	1	F18	UNCOATED	0,24	0.35	0.53	0.61	0.73
		1	1		F19	UNCOATED	0.21	0.39	0.59	0.75	0.94

⁽¹⁾ MEASUREMENTS ARE CHANGE IN LENGTH AFTER 5, 10, 15, 25, AND 50 ENTRY CYCLES.

⁽²⁾ SPECIMEN FAILURE (SF) OCCURRED ON 26TH CYCLE DUE TO ACCIDENTLY OVERLOADING IT TO 255 MN/m² (37 ksi) AT PEAK TEMPERATURE.

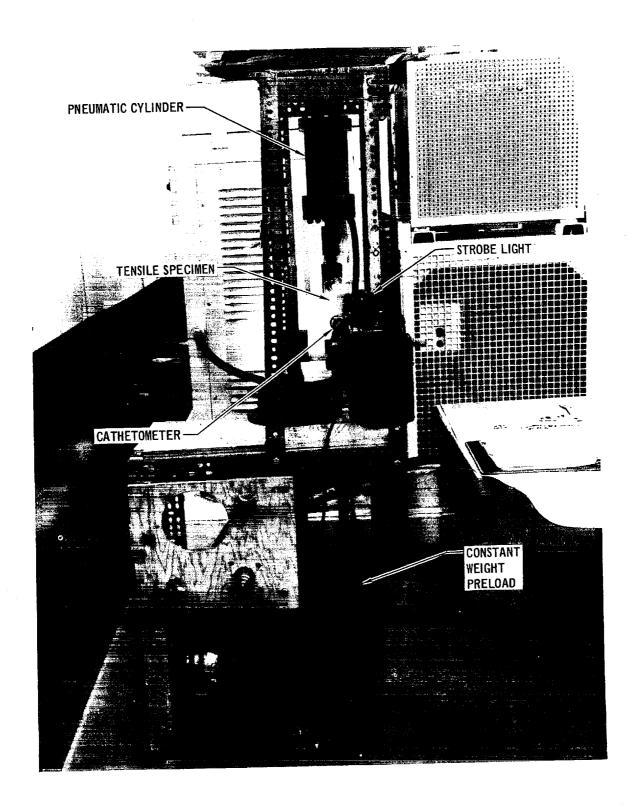
⁽³⁾ SPECIMEN REPLACES SPECIMEN L47 WHICH FAILED PREMATURELY.

In any case, it can be concluded that the creep properties of the alloys studied are unaffected by the presence of a salt coating. Furthermore, the magnitude of the creep deformations observed for these alloys is in agreement with earlier studies (Reference 21) which have shown that creep strains greater than those predicted from isothermal creep data can be obtained by cyclic stress and temperature conditions.

In addition to measuring the creep deformations, the crack length was measured after 5, 10, 15, 25 and 50 entry cycles. The crack length measurements were made by attaching the specimens to a pneumatic cylinder and then attaching weights to the specimens. The measuring equipment is shown in Figure 5-8. When the piston in the cylinder was raised, the weights were then lifted and the specimen was subjected to a constant load that was the equivalent of the final precrack stress shown in Table 3-4. The specimen was kept under this load for approximately five minutes while the crack length was measured using a cathetometer and strobe light. The strobe was found to be more desirable since it tended to highlight the cracks by pulsing the light.

The results of the crack length measurements are presented in Table 5-3. The inaccuracy of these measurements is evident if the changes in crack based on cathetometer measurements after 50 cycles are compared to these measured with the more accurate Scanning Electron Microscope after tensile test. Such inaccuracy can be attributed to difficulty in determining where the crack terminated. Frequently, in the case of the salt coated specimens, the cracks were known to have tunnelled under the salt deposits without cracking the deposits.

In the case of the columbium alloy FS-85, the specimen crack could not be observed directly because of the coating; however, the crack did propagate through the coating. The coating was suspected of cracking ahead of the base material because the ends of the cracks seemed jagged and appeared to follow the "grain boundaries" of the coating. 5-16



CRACK LENGTH MEASURING EQUIPMENT

Figure 5-8

Table 5-3 EFFECT OF ENTRY SIMULATION ON THE PRECRACK LENGTHS

Substituting analysis of a superior of a sup

>0	1	TEMP	STRESS	SS	SPECIMEN	NOILIGNO	INITIAL	CHANGE I	CHANGE IN CRACK LENGTH AFTER CYCLES $^{(1)}$ (mm)	ENGTH AF	TER CYCL	ES ⁽¹⁾ (mm)	FINAL	TOTAL
ALLO	8	9	MN/m ²	Ks.	NUMBER		LENGTH (mm)	5	10	15	25	SS	LENGTH ⁽²⁾ (mm)	EXTENSION (mm) (2)
INCONEL 718	811	1000	220	32	A10	SALT COATED	1.737	+0.262	+0.252	+0.335	+0.643	+1.161	3.218	+0.499
· ·					A29	UNCOATED	2.713	. 0.188 355	-0.135	+0.025	+0.152	+0,376	2.636	+0.269
	323	1200	165	24	A19	SALT COATED	1.481	-0.084	+0.348	+0.742	+0.528	+0.765	2.091	+0.610
					A50 A52	UNCOATED	2.271 2.870	+0.307	+0.569	+ 0. 358 +0.025	+0.381	+0.335	2.782 3.410	+0.511
RENE'41	1033	1033 1400	117	17	85 SE	SALT COATED SALT COATED	2.268	+0.343	+0.412	+0.440	+0.010	+0.462	2,575	+ 0.307
27 8 Valence de la constante				**********	RI3 RI5	UNCOATED	1.902	+0.768	+0.750	+0.760	+0.818	+0.775	2.674	+0.898
	1144	1600	28.6	.5 .5	R1	SALT COATED	2,096	+0.525	+0.411	+0.381	+0.256	+0.213	2.554	+0.458
					R16 R17	UNCOATED UNCOATED	2.604	+0.160	+0.269	+0.269	+0.299	+0.182	2.255 2.875	-0.349
5097	1144	1600	34.5	5.0	L15 L46	SALT COATED	1.864	+0.765	+0.760	+0.821	+0,889	+1,021	2,633	+0.769 +0.346
					로 중	UNCOATED	2.436	+0.206	+0.206	+0.302	+0.383	+0.411 +0.419	2.560 2.822	+0.124 +0.340
	1255	1255 1800	13.8	2.0	L16	SALT COATED	1,994	+0.631	+0.693	+0,790	+0.780	+1,161	2.632 2.750	+0.638
ą.					L56 L57	UNCOATED	2.441	+0.274	+0.437	+0.424	+0.480	+0.503	2.729 2.680	+0.288
HASTELLOY X 1255	1255	1800	6.9	1.0	ZX	SALT COATED	2.446	+0.854	+0.744	+0.587	+0.559	+0.572	2.998	+0,552
	4. 7.35 N. 266			1274	A TONE			Contraction of						20 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 2 1 1 1 2 1 1 1 2 1 1 1 2 1 1 1 1 2 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1

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FOLDOUT FRAME

MCDONNELL

16 - Hilliam Barrier March			•			**			110		ill Dept.			* 4
	1	T			RES	UNCOATED	1.902	+0.768	+0.750	+0.760	+0.818	+0.775	2.800	+0.838 +
					R15	UNCOATED	2.230	+0.635	+0.592	+0.620	+0.480	+0.39	2,674	+ O. 444
		-				CALT COATED	2 096	+0 525	+0.411	+0.381	+0,256	+0.213	2,554	+0.458
	¥ 1	1600	28.6	ά.Σ	12 6	SALI CONTED	2020	+0.054	100+	+0.219	+0.343	+0.280	2.360	+0.290
				_	717	INCOATED	2 63 6	+0.160	+0.269	+0.269	+0.299	+0.182	2,255	-0.349
					2 2	UNCOATED	2,362	+0.277	+0.277	+0.549	+0.496	+0.470	2,875	+0.513
					1	400	1 054	70.0	US 2 0 T	+0 821	+0.889	+1.021	2.633	+0.769
L605	1144	<u>6</u>	34.5	2.0	í.	SALI COATED	1,004	178	10,700	10.04	+0.366	+0.325	2.896	+0.346
					₹ :	SALI CUATED	966.2	90770	50°0+	+0.302	+0.383	+0.411	2,560	+0.124
-					<u> </u>	UNCOATED	2.482	050"0+	+0.302	+0.381	+0.378	+0.419	2,822	+0.340
					3		700	163.0	TO 503	V02 UT	180 T	+1161	2.632	+0,638
	1255	1255 1800 13.8	13.8	2.0	917	SALICUALED	1.934	10.031	1 202	06/*n	2001	700.0	2.750	-0.062
					<u>S</u>	SALT COATED	2.812	+0.101	+1.332	70.70	20.04	+0.63	2 729	+0.288
					ا ا ک	UNCOATED	720	+0.2/4	+0.43/	+0.424	020-0+	+0.020	2.680	-0.040
					<u>ج</u>	UNCOALED	07/57	7 .1. 0	20170					
UACTELL DV V 1255 1200	1255	1 200	6 4	-	12X	SALT COATED	2,446	+0.854	+0.744	+0.587	+0.559	+0.572	2.998	+ 0.552
HASI ELLOI A	M77	3	}	?		SALT COATED	2,436	+0.312	-0.127	+0.330	+0,386	+0.350	2.770	+0.334
					×15	IINCOATED	2 718	+0.025	+0.040	+0.058	+0.074	-0.045	1.670	-1.048
					736 X36	INCOATED	2,479	+0.777	+0.785	+0.871	+0.638	+0.640	2.733	+0.254
بندر					3			6					,	1000
-	1366	2002	3.5	0.5	X28	SALT COATED	2.139	(c)WN	+0.183	-0.1 %	중 주	+0.059	1.508	1.031
	}		}	}	X35	SALT COATED	1.991	+0,684	+0.719	+0.666	+0.110	+0.643	2.4/5	+0.484
53					X39	UNCOATED	2.197	+0.155	+0.168	+0.142	+0.198	+0.168	2.235	+0.038
i di					X41	UNCOATED	2.436	+0.005	Σ	-0.757	-0.7 87	-1.166	3.626	1.19
	;	į		-	Ç	CALTOOATED	2 065	Z	£2	-0.137	ž	-0.612	2,168	+0.103
TONIC.	1366	2000 34.5	34.5	5.0	77	SALI COATED	2002	0 025	+0.056	-0.173	P 033	+0.018	3.125	+0.425
_					123	SALI CUATED	20.7		10.03	0113	196	787	2 460	-0.121
•					170	UNCOATED	196.7	+0.03+	7 7	מולים היים היים	2770	795 0+	3.575	+0.632
					T35	UNCOATED	2.833	+0.384	+0.30+	00.01	Ì.			
	1.477	22000 24 1	24.1	6	F	SALT COATED	3,566	-1.567	-0.574	-0.8 10	-1.051	-0.632	4.164	+0.598
	<u>}</u>		7-4-7	r,	125	SALT COATED	3.063	-0.612	-0.528	9.50	-0.365	-0.320	2.902	-0.161
					15	UNCOATED	2.929	-0.884	797.0	-0.435	-0.3 61	-0.653	2.560	-0,369
					123	UNCOATED	3.086	-0.825	-1.227	-1.077	95° 9	-0.825	2,500	1.386
	,			,	: 6	CALT COATEN	2.250	813		-0.302	-0.533	Ž	3,291	+1.041
R512E/FS85	14//	14// [2200 68.9		=	e e	SALI CONTED	2 263	303	9	73%	-0.130	ž	2.656	+0,393
					ខ្ម	SALI COALED	020 0	10.04	+0 234	+0 338	+1.801	ž	3,698	+0.759
ý e man					3 5	UNCOALED	2,293	+0.012	+0.424	-0.143	60.0	¥	3,700	+1,411
paraman						CHOOSE STATE		. 6 503	132	+ 1 306	+3.122	Z	2,785	+0.123
	158	1589 2400 44.8	4 .8	6.5		SALI CUALED	799.7	TU.332	T 0007	+1 047	+ 2 685	Ž	4.222	+1.375
					F12	SALI CUATED	7.84/	+0./30	000	± 1.476	+2 446	Z	4.060	+1.395
					F 18	UNCOATED	2.360	+0.43/		+1.903	+ 3.886	E N	4.379	+2.019
	4	_		4		a lyong								

 $^{(1)}$ CRACK LENGTH DETERMINED AFTER APPROPRIATE NUMBER OF CYCLES $^{(2)}$ DETERMINED ON TENSILE TESTED SPECIMEN WITH THE SCANNING ELECTRON MICROSCOPE. $^{(3)}$ NOT MEASURED

FOLDOUT FRAME

I Methodological at 1 1 1

5–18

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The way to be the first

The cracks in the Hastelloy X specimens tended to open and blunt, but this could be a result of the higher percentage of creep that was observed in the alloy. The blunting of the crack was not observed in any other alloy tested.

The cracks in the Inconel 718 specimens were particularly difficult to measure because a line appeared to run from the crack to the edge of the specimen. This line confused measurements and introduced errors into the determination of the crack lengths.

In addition to the above problems with the individual alloys, some cracks were easier to measure than others within a specific group. The variations within a group were probably due to oxidation around the crack tip, which tended to obscure the crack. In the FS-85 alloy, the holes that were drilled through the coating showed progressive attack by oxidation with increasing cycles. This is a characteristic trait of columbium alloys which require oxidation protective coatings above 811°K (1000°F). Any defect in the coating allows oxygen to enter and, with time, embrittles the substrate. Crack length measurements were not made on the FS-85 after the 50th cycle because the specimens were embrittled by oxygen contamination from the defect, and one failed prematurely during the crack measurement.

The negative values of crack extension that appear in Table 5-3, therefore, are a direct indication of the inaccuracy of the cathetometer crack length measurements and are not a result of a crack healing process. These data are, then, useful for comparative purposes only and not for any fracture mechanics calculations. The data does indicate that crack propagation for these alloys is insensitive to the presence of salt coatings.

In addition to measuring the change in crack length and the creep of the specimens, periodic visual examinations were made of the specimens. Visual examination revealed that variations existed between some of the salt coated

specimens and the uncoated specimens. The salt coated specimens of Hastelloy X, for example, had a green tinge while the uncoated specimens were grey in color. The coated FS-85 specimens tested to 1477°K (2200°F) showed a difference between the salt coated and uncoated specimens. The uncoated specimens showed color banding in the gage length while the salt coated specimens did not. The discoloration was only evident for the first fifteen entry cycles, and then both the salt coated and uncoated specimens turned a dark brown color. The same effect was noted on the FS-85 specimens tested at 1589°K (2400°F) except a shorter number of cycles resulted in a brown color. The salt coated TDNiCr specimens were lighter colored in the gage length compared to the uncoated specimens while the L-605 and Rene' 41 specimens showed no clear color changes, but after the specimens were cycled and cleaned, a white residue was left on the salt coated specimens. Of all the alloys tested, only the Inconel 718 showed no discoloration or residue after cycling.

Since all the tensile specimens subjected to elevated temperature testing remained intact after 50 simulated entry cycles, room temperature tensile tests were conducted to determine residual strength. These data are presented in Table 5-4. Comparison of the tensile properties of salt coated and uncoated specimens shows that the presence of a salt coating during simulation entry cycling has no significant effect on the residual tensile strength and elongation of each alloy. An exception appears to be the Hastelloy X samples cycles to 1366°K (2000°F), for which the tensile strength of the salt coated specimens is lower than that of the uncoated specimens. Subsequent metallographic examination, discussed in greater detail below, showed that the degradation in strength was due to localized thermal spikes within the furnace on this particular test rather than to hot corrosion effects.

The data in Table 5-4 also indicate that the tensile properties of these alloys are unaffected by the simulated reentry testing, since their ultimate

Table 5-4

EFFECT OF ENTRY SIMULATION ON THE ROOM TEMPERATURE NOTCHED TENSILE PROPERTIES OF PRECRACKED HIGH TEMPERATURE ALLOYS

ALLOY	CY	IMUM Cle Rature	MAXI NET C STR	YCLE	F _{tn} BEFORE C	YCLING		F _I AFTER 5	0 CYCLE		FAILING ELONGATION BEFORE CYCLING,	(CREE	NGATION P) AFTER YCLES, %	ELOI AFTER	AILING NGATION 50 CYCLES, RCENT
	σK	°F	MN/m²	KSI	MN/m²	KSI	WITH MN/m ²	SALT KSI	WITHOU MN/m ²	JT SALT KSI	PERCENT	WITH SALT	WITHOUT SALT	WITH SALT	WITHOUT SALT
INCONEL 718	811	1000	221.0	32.0	1220 1320 AVG: 1270	192	1290 1260 1280	187 183 185	1260 1260 1260	183 183 183	2.0 1.5 1.8	0.56 0.42 0.49	0.45 0.47 0.46	2.8 2.5 2.6	6.1 3.1 4.6
	922	1200	165.0	240.0	AVG:		1200 1290 1240	174 187 180	1280 1260 1270	185 183 184		0.48 0.39 0.44	0.49 0.44 0.46	0.4 2.2 1.3	2.8 2.1 2.4
RENE'41	1033	1400	117.0	17.0	814 869 AVG: 841	126	945 931 938	137 135 136	889 931 910	129 135 132	0.5 0.5 0.5	1.04 0.36 0.70	0.73 0.26 0.50	12.5 11.8 12.2	1.0 1.2 1.1
	1144	1600	58.6	8.5	AVG:		841 800 820	122 116 119	745 807 776	108 117 112		0.13 0.04 0.08	0.04 0.42 0.23	1.2 2.5 1.8	1.8 1.5 1.6
L-605	1144	1600	34.5	5.0	662 674 AVG: 669	96.0 97.8 96.9	596 664 630	86.4 96.3 91.4	655 585 620	95.0 84.8 89.9	3.5 3.5 3.5	0.11 0.18 0.14	0.19 0.43 0.31	3.0 5.0 4.0	3.9 5.1 4.5
	1255	1800	13.8	2.0	AVG:		612 639 626	88.7 92.7 90.7	670 661 666	100.0 95.8 97.6		0.40 0.74 0.57	0.71 0.42 0.56	2.4 4.1 3.2	5.0 5.0 5.0
HASTELLOY X	1255	1800	6.89	1.0	585 600 AVG: 592	84.8 87.0 85.9	580 592 586	84.1 85.9 85.0	534 601 568	77.5 87.2 81.4	3.0 2.5 2.8	0.20 0.49	0.75 0.65 0.70	2.3 5.1 3.7	5.2 4.6 4.9
	1366	2000	3.45	0.5	AVG:		419 438 428	60.7 63.5 62.1	466 594 530	67.6 86.1 76.8		0.50 0.48 0.49	1.22 1.09 1.16	9.0 2.7 5.8	7.5 6.4 7.0
TDNiCr	1366	2000	34.5	5.0	654 635 AVG: 644	94.8 92.1 93.4	644 665 654	93.4 96.5 95.0	634 648 641	92.0 93.8 92.9	0.5 0.5 0.5	0.43 0.30 0.36	0.89 0.13 0.51	1.9 2.2 2.0	1.9 1.5 1.7
	1477	2200	24.1	3.5	AVG:		662 614 638	96.0 89.0 91.5	618 659 638	89.6 95.6 92.6		0.81 0.26 0.54	0.45 0.78 0.61	2.2	1.8 3.2 2.5
R-512E/FS-85	1477	2200	68.9	10.0	592 611 AVG: 602	85.9 88.6 87.2	487 407 447	70.7 59.0 64.8	313 313	45.4 45.4	3.5 2.5 3.0	0.61 0.44 0.52	0.33 0.43 0.38	2.1 2.5 2.3	1.6 3.0 2.3
	1589	2400	44.8	6.5	AVG:		303 237 270	43.9 34.4 39.2	392 300 346	56.9 43.5 50.2		0.82 0.68 0.75	0.73 0.94 0.84	1.4 2.0 1.7	2.0 0.4 1.2

^{*}FAILED ON LOADING

^{**}BROKE OUTSIDE GAGE

FS-85 specimens, an exception, have undergone a degradation in tensile strength and elongation, attributable to oxygen contamination near the crack. This is in agreement with the results obtained under a separate study (Reference 17), which also tested precracked R512E coated FS-85 specimens.

After tensile testing, the fracture surfaces of the specimens were examined and photographed with the Scanning Electron Microscope to analyze failure mode and to determine the extent of crack propagation during simulated entry testing. These photographs, shown in Appendix B, show the same transition from fatigue precracking to tensile failure as found in the fractographs of the alternate immersion specimen of Figure 4-4. For each alloy, the fracture surface of the salt coated specimen is indistinguishable from that of its companion uncoated specimen. With the exception of the R512E coated FS-85, all of the tensile failure zones are of the dimple rupture type which is characteristic of a ductile material. In contrast, the fractographs of the FS-85 specimens reveal that only part of the tensile failure zone contains evidence of a dimpled rupture failure, the rest having the faceted appearance of a brittle cleavage fracture. These faceted regions, only observed in the area adjacent to the crack tip, indicate that oxygen contamination has occurred for some distance ahead of the crack.

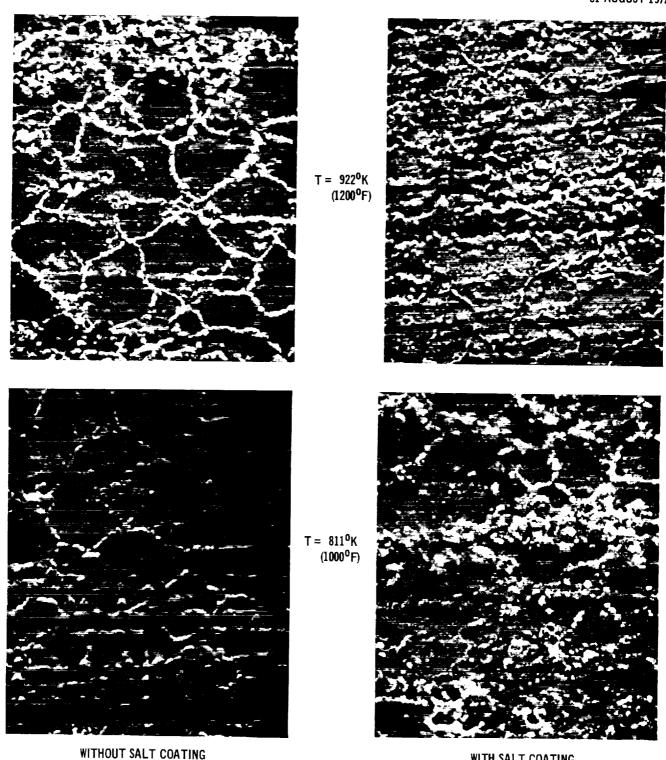
Weight change measurements were made on the control specimens after 25 and 50 cycles. Prior to these measurements, the controls were cleaned by placing them in hot water and then wiping them with cheesecloth. This was followed by cleaning in acetone and compressed air. The coated FS-85 specimens were not wiped with cheesecloth because of the coating roughness, which might have entrapped pieces of cheesecloth in the surface of the specimens, causing errors in the weight measurements. The results of the weight change measurements, shown in Table 5-5, indicate that all specimens experienced a net weight gain, primarily due surface oxidation.

Table 5-5
CHANGE IN WEIGHT OF CONTROL COUPONS
AFTER ENTRY SIMULATION

ALLOY	TEMPE	RATURE	CONDITION	WEIGHT CHANGE AFTER	WEIGHT CHANGE AFTER	
ALLUT	٥K	0F	CONDITION	25 CYCLES (mg)	50 CYCLES (mg)	
INCONEL 718	811	1000	SALT COATED UNCOATED	-0.4 -0.7	0.0 -1.6	
	922	1200	SALT COATED UNCOATED	-0.4 -0.7	+0.7 -0.7	
RENE' 41	1033	1400	SALT COATED UNCOATED	+1.9 -0.6	+ 0.8 -0.7	
	1144	1600	SALT COATED UNCOATED	+1.5 0.4	+2.4 +1.4	
L605	1144	1600	SALT COATED UNCOATED	0.0 0.3	+ 0.8 + 0.0	
	1255	1800	SALT COATED UNCOATED	+4.1 +2.2	+ 4.8 + 2.8	
HASTELLOY X	1255	1800	SALT COATED UNCOATED	+ 4.0 + 0.9	+ 5.5 + 2.3	
	1366	2000	SALT COATED UNCOATED	+0.3 +7.0	+1.3 +11.4	
TDNiCr	1366	2000	SALT COATED UNCOATED	0.0 +0.3	+1.7 +0.6	
	1477	2200	SALT COATED UNCOATED	+6.5 +3.1	+7.8 +3.7	
R512E/FS-85	1477	2200	SALT COATED UNCOATED	+58.8 +19.0	+62.3 +33.8	
	1589	2400	SALT COATED UNCOATED	+ 52.4 + 33.8	+63.8 +36.4	

457-3066

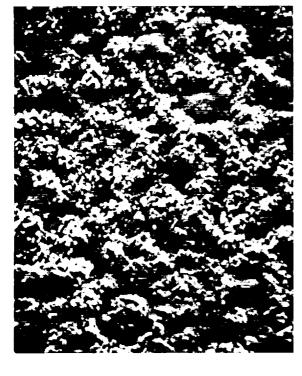
The fact that the salt coated specimens gained more weight than the uncoated specimens is in most cases due to the presence of residual salt particles not removed during the cleaning process. This conclusion is confirmed by the Scanning Electron Microscope photographs of Figure 5-9, showing the condition of the coupon surfaces after 50 simulated entry cycles. Besides confirming the presence of salt particles, these photographs also show that the surface of each salt coated specimen is nearly identical to that of its companion uncoated specimen. An exception



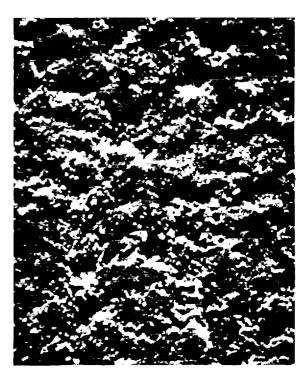
SCANNING ELECTRON MICROSCOPE PHOTOGRAPHS OF THE SURFACES OF INCONEL 718 TENSILE SPECIMENS AFTER 50 SIMULATED ENTRY CYCLES (1000X)

457-3067

WITH SALT COATING



 $T = 1144^{\circ} K$ (1600°F)



 $T = 1033^{\circ} K$ $(1400^{\circ} F)$



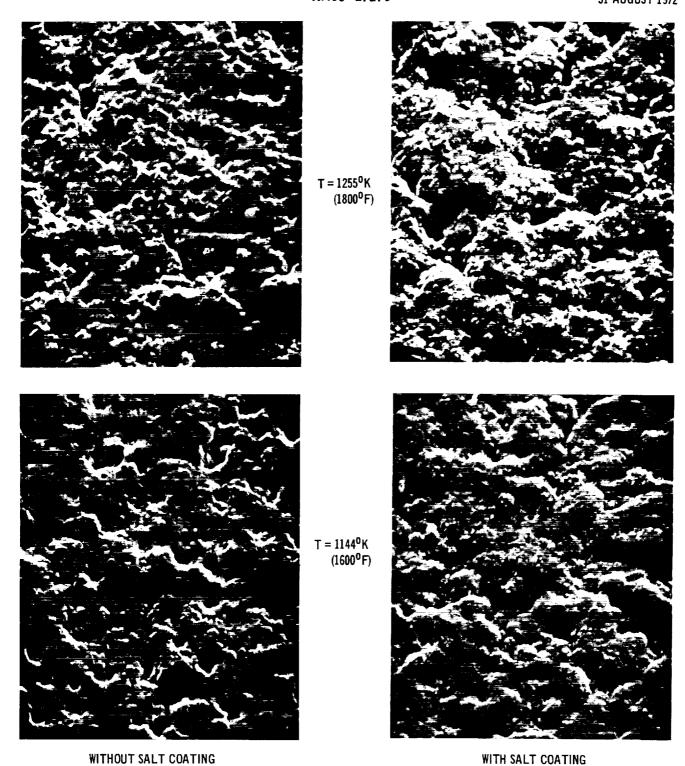
WITHOUT SALT COATING

WITH SALT COATING

SCANNING ELECTRON MICROSCOPE PHOTOGRAPHS OF THE SURFACES OF RENE '41 TENSILE SPECIMENS AFTER 50 SIMULATED ENTRY CYCLES (1000X)

457-3068

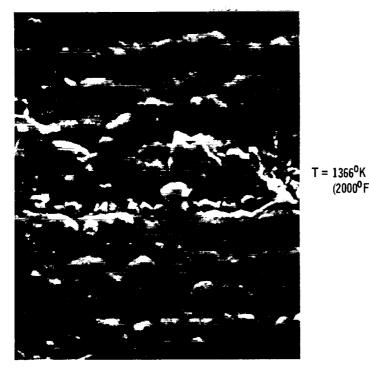
Figure 5-9 (b)

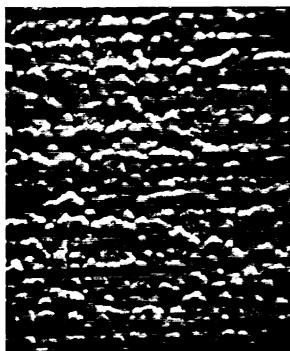


SCANNING ELECTRON MICROSCOPE PHOTOGRAPHS OF THE SURFACES OF L-605 TENSILE SPECIMENS AFTER 50 SIMULATED ENTRY CYCLES (1000X)

Figure 5-9 (c)

(2000°F)







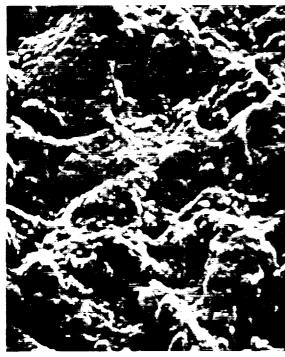
WITHOUT SALT COATING

WITH SALT COATING

SCANNING ELECTRON MICROSCOPE PHOTOGRAPHS OF THE SURFACES OF HASTELLOY X TENSILE SPECIMENS AFTER 50 SIMULATED ENTRY CYCLES (1000X)Figure 5-9 (d)

457 - 3070

 $T = 1255^{0}K$ $(1800^{\circ}F)$



T = 1477^oK (2200^oF)



 $T = 1366^{\circ} K$ (2000°F)



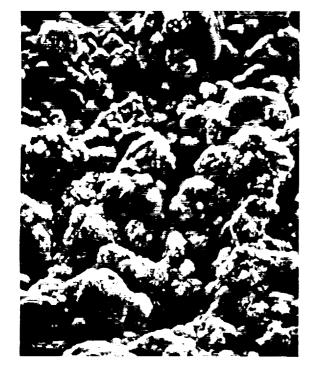
WITH SALT COATING

WITHOUT SALT COATING

SCANNING ELECTRON MICROSCOPE PHOTOGRAPHS OF THE SURFACES OF TDNiCr TENSILE SPECIMENS AFTER 50 SIMULATED ENTRY CYCLES (1000X)

457-3071

Figure 5-9 (e)



 $T = 1589^{0} K$ (2400°F)





WITHOUT SALT COATING

 $T = 1477^{0}K$ $(2200^{0}F)$



WITH SALT COATING

SCANNING ELECTRON MICROSCOPE PHOTOGRAPHS OF THE SURFACES OF R-512E COATED FS-85 TENSILE SPECIMENS AFTER 50 SIMULATED ENTRY CYCLES (1000X)

Figure 5-9 (f)

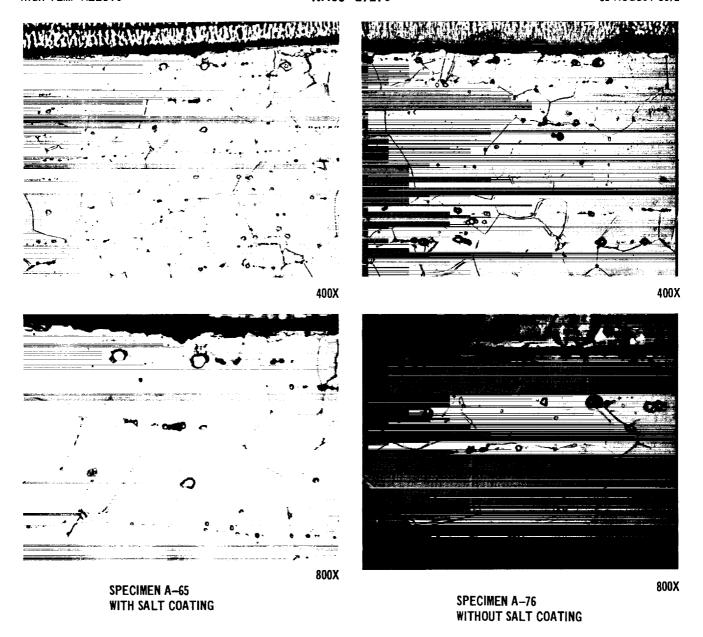
457-3072

is the condition of the R512E coated FS-85 specimen tested at 1589°K (2400°F) with a salt coating. A fusion reaction appears to have taken place, since the surface of this specimen appears much smoother than that of its uncoated companion. It is considered that such a reaction is primarily due to the interaction of NaCl with the oxidized constituents of the R512E coating, which has a nominal composition of Si-20Cr-20Fe. The presence of NaCl serves as a flux to form a low melting vitreous coating containing sodium and silicon, similar to that obtained by salt glazing clayware. The absence of this glaze on the surface on the salt coated specimen cycled to the lower temperature (1477°K) is perhaps an indication of the temperature/time sensitivity of the reaction.

The microstructures of the control coupons after 50 cycles of simulated entry testing are shown in Figures 5-10 to 5-16. For all alloys comparison of the microstructures of salt coated and uncoated specimens shows that no degradation has occurred as a result of the salt coating. Comparison of the alloy microstructures before (Figure 3-2) and after exposure shows that only the Inconel 718 specimens (Figure 5-10) have not been affected. Although the same was observed for the base material in the FS-85 specimens shown in Figure 5-11, the degree of cracking in the R512E coating increased, presumably as a result of thermal fatigue.

Examination of the microstructures of both salt coated and uncoated TDNiCr specimens in Figure 5-12 shows the presence of void formations that were absent in the material before test. The voids in the specimens cycled to 1477°K (2200°F) are large and evenly distributed, whereas those in the specimens cycled to 1366°K (2000°F) are small and concentrated near the surface. Such void formation, observed in an earlier study (Reference 22), is considered to result from Kirkendall diffusion of chromium. The evaporation of chromium oxide from the surface produces the necessary chemical gradient to cause the internal voids by Kirkendall diffusion processes. Such an explanation would account for the temperature dependence of the

457~3073



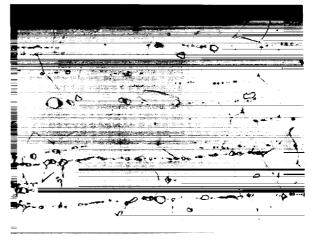
MICROSTRUCTURES OF INCONEL 718 SPECIMENS AFTER 50 SIMULATED

ENTRY CYCLES AT 811°K (1000°F)

Figure 5-10 (a)

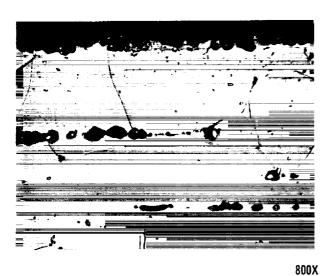
size and distribution of the voids in Figure 5-12. Close examination of the voids in these photographs show that a number of them appear to have small particles in their centers. Such particles have been identified as chromium oxide (Reference 22), and serve as points of precipitation of vacancies that lead to the void formations.





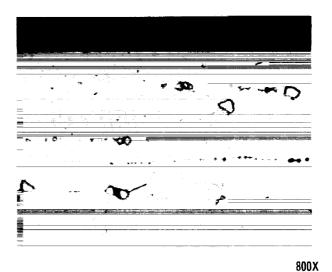
400 X

400 X



SPECIMEN A-69 WITH SALT COATING

457-3074



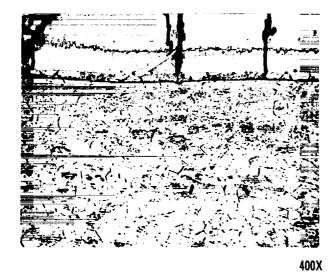
SPECIMEN A-75
WITHOUT SALT COATING

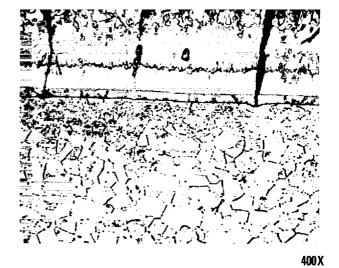
MICROSTRUCTURES OF INCONEL 718 SPECIMENS AFTER 50 SIMULATED ENTRY CYCLES AT 922°K (1200°F)

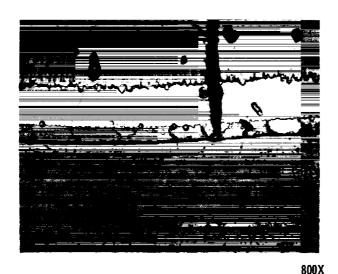
Figure 5-10

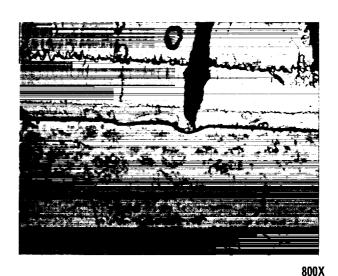
(b)

For the L-605 specimens shown in Figure 5-13, the extent of twinning has been drastically reduced as a result of the thermal exposure, and some coarsening of the grain boundary carbides has occurred. Some subsurface depletion of these car-









SPECIMEN F-80
WITH SALT COATING

457-3075

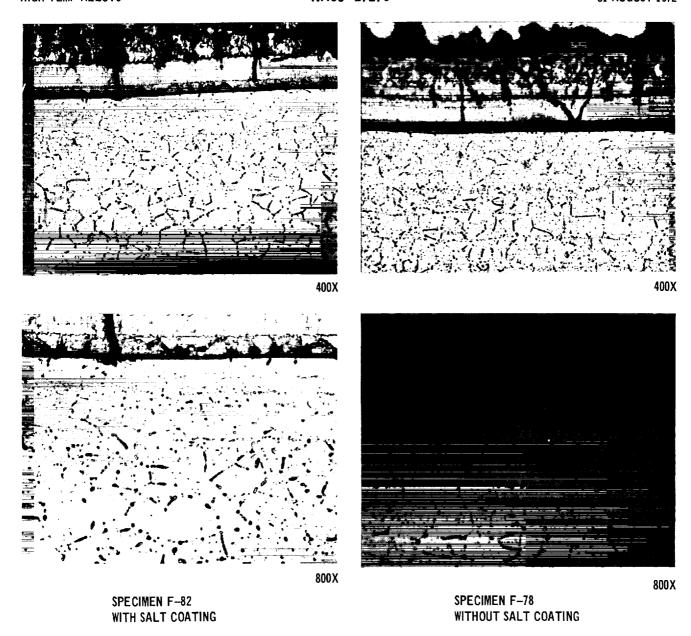
SPECIMEN F-76
WITHOUT SALT COATING

MICROSTRUCTURES OF R-512E COATED FS-85 SPECIMENS AFTER 50 SIMULATED ENTRY CYCLES AT 1477°K (2200°F)

Figure 5-11 (a)

bides has occurred for all L-605 specimens because of oxidation effects. It is to be noted that the specimens without a salt coating exhibit slightly more surface depletion than the salt coated specimens. This behavior can be attributed to the fact that the salt coating is actually providing surface protection from the oxygen environment at these temperatures. 5-33

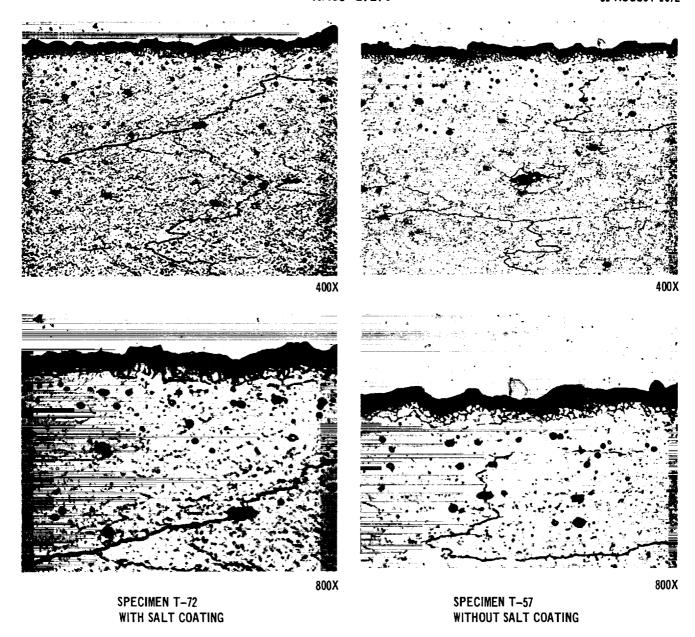
457-3076



MICROSTRUCTURES OF R-512E COATED FS-85 SPECIMENS AFTER 50 SIMULATED ENTRY CYCLES AT 1589°K (2400°F)

Figure 5-11 (b)

Similar but more pronounced microstructural phenomena are observed in the Hastelloy X specimens shown in Figure 5-14. All specimens exhibit a subsurface zone depleted of precipitates. For the specimens cycled to 1255°K (1800°F), this depleted zone is greater for the uncoated specimen than for the coated specimen,

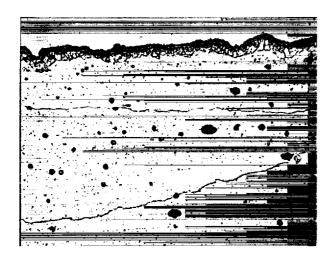


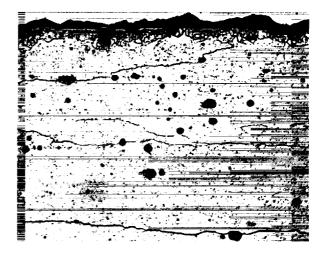
MICROSTRUCTURES OF TDNiCr SPECIMENS AFTER 50 SIMULATED ENTRY CYCLES AT 1366°K (2000°F)

457-3077

Figure 5-12 (a)

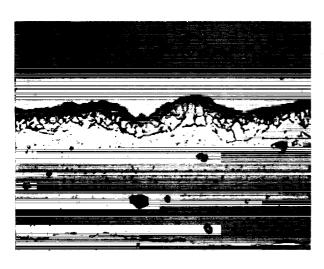
again indicating the slightly protective nature of the salt coating. Extensive carbide agglomeration has also occurred in all specimens, with the exception of the salt coated sample cycled to 1366°K (2000°F). For this specimen, grain growth and dissolution of carbide precipitates can be observed along with recrystallization of

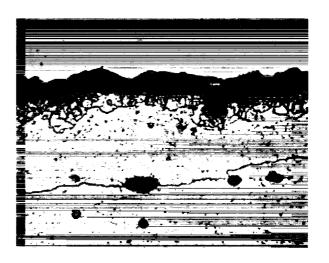




400 X

400 X





800X

800X

SPECIMEN T-73 WITH SALT COATING

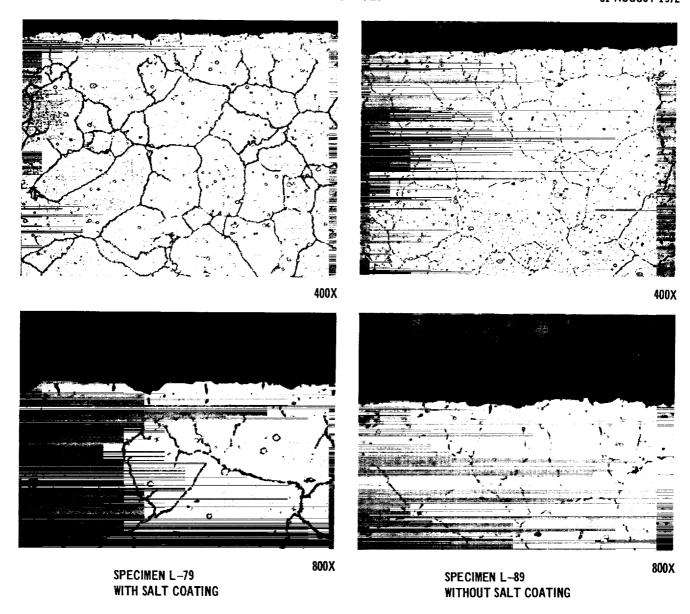
457-3078

SPECIMEN T-59
WITHOUT SALT COATING

MICROSTRUCTURES OF TDNiCr SPECIMENS AFTER 50 SIMULATED ENTRY CYCLES AT 1477°K (2200°F)

Figure 5-12 (b)

the subsurface layer. Since examination of the salt coated tensile specimens cycled at this temperature shows that similar grain growth occurred in isolated regions along the specimen length, it is felt that such microstructural changes are due to



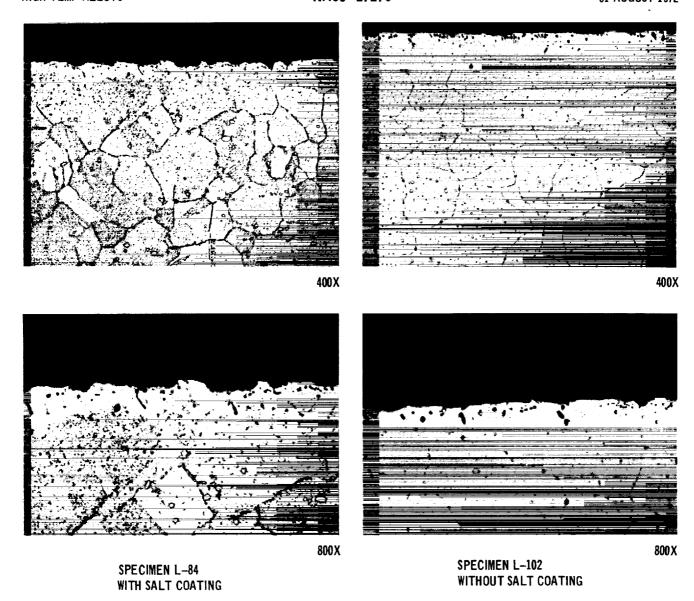
MICROSTRUCTURES OF L-605 SPECIMENS AFTER 50 SIMULATED ENTRY CYCLES AT 1144°K (1600°F)

457-3079

Figure 5-13 (a)

localized high temperature regions within the furnace of the reentry simulator. This effect is substantiated by the results of a thermal stability study on this alloy (Reference 23), which found that agglomerated carbides go into solution within one hour at 1421°K (2100°F). Such microstructural changes could account for the somewhat lower tensile strengths reported in Table 5-4 for the salt coated specimens cycled to 1366°K (2000°F).

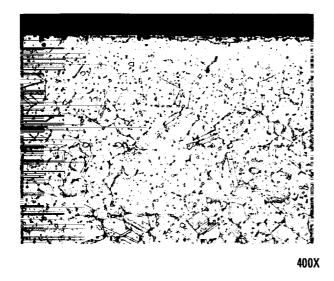
457-3080



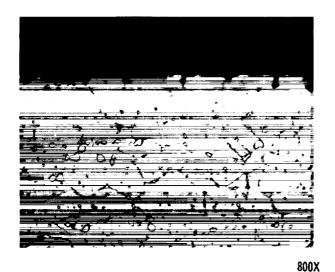
MICROSTRUCTURES OF L-605 SPECIMENS AFTER 50 SIMULATED ENTRY CYCLES AT 1255°K (1800°F)

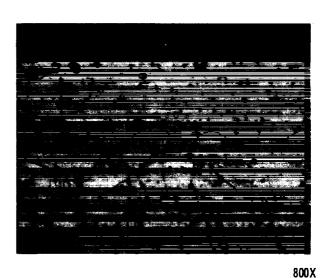
Figure 5-13 (b)

The electron microprobe analyzer was used to try to quantify the extent of subsurface reaction in both salt coated and uncoated Hastelloy X specimens cycled to 1144°K (1600°F). These results, shown in Figure 5-15, show that a chromium-rich oxide has formed on the surface of specimens, while the subsurface layer has become depleted of this element. Such chromium depletion is a result of the manner in which oxidation occurs, since a study (Reference 24) determined the oxide









SPECIMEN X-93
WITH SALT COATING

SPECIMEN X-81
WITHOUT SALT COATING

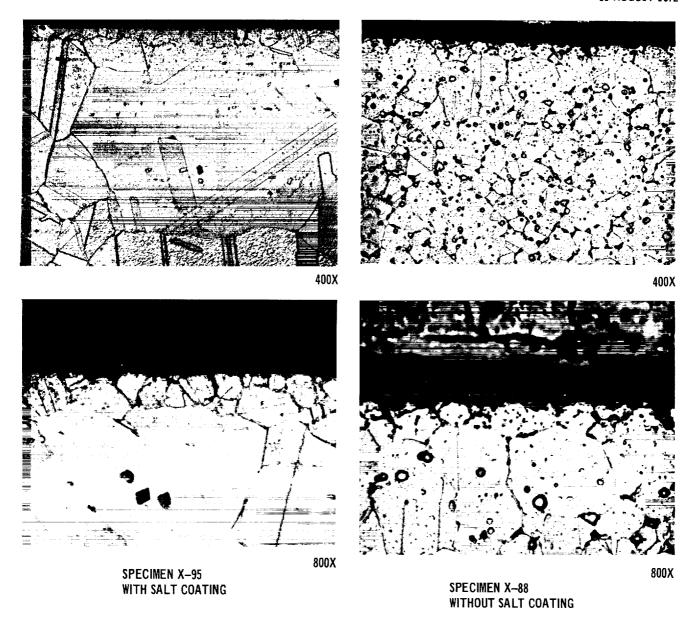
MICROSTRUCTURES OF HASTELLOY-X SPECIMENS AFTER 50 SIMULATED ENTRY CYCLES AT 1255°K (1800°F)

457-3081

Figure 5-14 (a)

composition of Hastelloy X oxidized at 1144°K (1600°F) to be predominately NiCr₂O₃. Also to be noted from Figure 5-15 is the fact that the degree of chromium segregation is slightly less for the salt coated specimen than for the uncoated specimen, confirming that the salt coating is providing some protection from the oxidative environment.

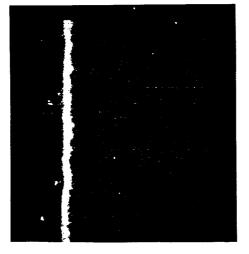
457-3082



MICROSTRUCTURES OF HASTELLOY X SPECIMENS AFTER 50 SIMULATED ENTRY CYCLES AT 1366°K (2000°F)

Figure 5-14 (b)

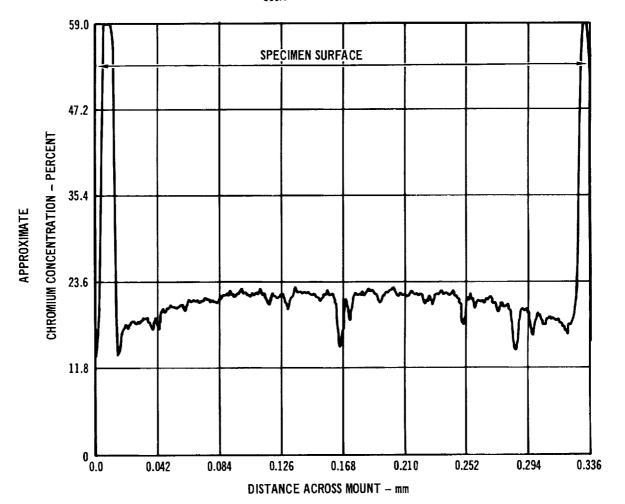
The microstructures of the Rene' 41 specimen after entry simulation are shown in Figure 5-16 and also show that pronounced changes have occurred as a result of the exposure. Extensive carbide agglomeration can be observed in each specimen, while the extent of the subsurface depleted layer had increased over that present in uncycled material (Figure 3-2). Microprobe scans across the thickness of salt coated and uncoated specimens exposed at 1144°K (1600°F) are shown in Figures 5-17





800X

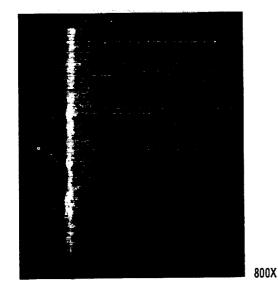
800X



CHROMIUM X-RAY RASTER IMAGE AND MICROPROBE SCAN FOR HASTELLOY X SPECIMEN X-88 AFTER 50 SIMULATED ENTRY CYCLES AT 1366°K (2000°F), WITHOUT SALT COATING

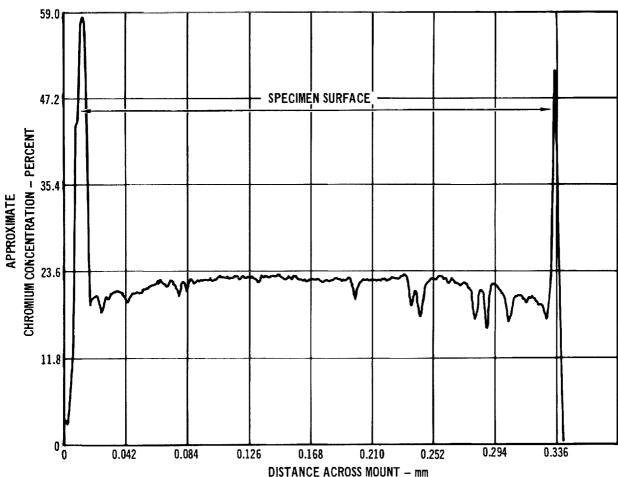
457-3083

Figure 5-15(a)





800X



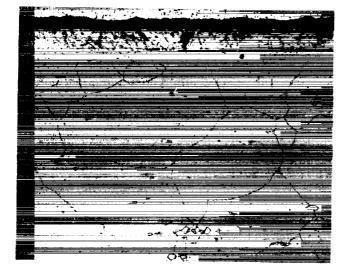
CHROMIUM X-RAY RASTER IMAGE AND MICROPROBE SCAN FOR HASTELLOY X SPECIMEN X-95 AFTER 50 SIMULATED ENTRY CYCLES AT 1366°K (2000°F, WITH SALT COATING

Figure 5-15 (b)

and 5-18. These results indicate that the salt coated specimens are somewhat less affected by segregation than the uncoated specimens. Furthermore, the subsurface layer is depleted of chromium and aluminum, with discrete titanium-rich area. As in the case of the Hastelloy X alloy, the depletion of alloying elements in the subsurface layer is due to preferential oxidation of the less-noble alloying constituents, particularly aluminum and titanium. A study which investigated this behavior, Reference 25, found that the initial oxidation of Rene' 41 involves the formation of a film of α -Al₂O₃. Subsequent oxidation allows chromium ion diffusion, converting the oxide film into a (Al, Cr)₂O₃ solid solution, while prolonged exposure was found to produce overgrowths of TiO₂. The subsurface acticular phase observed in Figure 5-17 was identified in this study as TiN.

It is to be noted that the results of this study do not appear to agree with those obtained in Reference 11, discussed in detail in Section 2.3. Briefly, this study found that 0.6 mm (.015 inch) salt coatings degraded the room temperature mechanical properties of Inconel 718, Rene' 41, and TDNiCr after each was exposed to elevated temperatures for 48 hours in air. However, it was also found that this degradation was sensitive to the partial pressure of oxygen during the exposure. No significant degradation was observed when the exposure was conducted in an argon atmosphere, and only slight degradation was noted for a 1% oxygen - 99% argon test environment, which in this case corresponds to an oxygen partial pressure of 1010 N/m² (7.60 Torr).

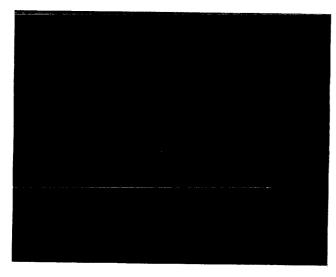
This apparent anomaly is resolved when consideration is given to the thickness of the salt coating used in the present study. As discussed in Section 2.2, (Reference 9), thick salt deposits on Inconel 718 specimens caused greater mechanical property degradation than thin coatings. On this basis the .025 mm (.001 inch) thick coatings used in the present study would be expected to produce less degradation in the post exposure mechanical properties than the 0.6 mm (.015 inch)

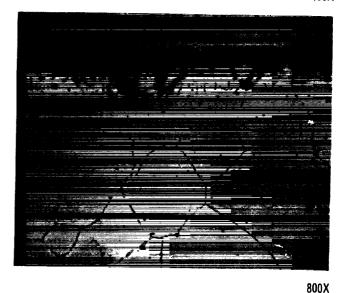




400X

400X





800X

SPECIMEN R-81 WITH SALT COATING

SPECIMEN R-70 WITHOUT SALT COATING

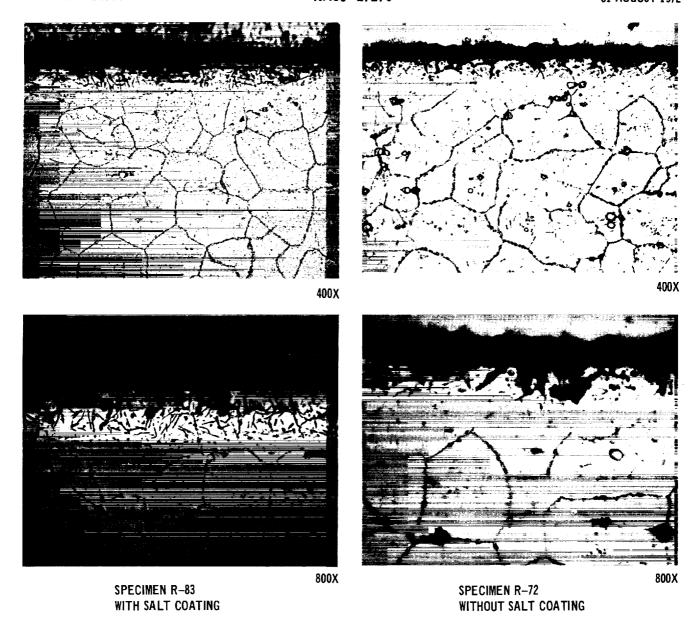
MICROSTRUCTURES OF RENE 41 SPECIMENS AFTER 50 SIMULATED ENTRY CYCLES AT 1033°K (1400°F)

457-3085

Figure 5-16

(a)

deposits used in the study cited in Reference 11. However, the extent to which this factor is responsible for the differences in the two sets of test data cannot be determined because of additional differences in environmental pressures used during exposure.

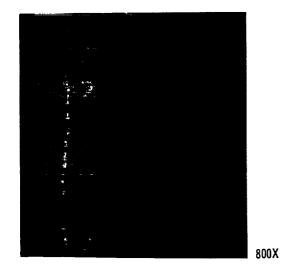


MICROSTRUCTURE OF RENE '41 SPECIMEN AFTER 50 SIMULATED ENTRY CYCLES AT 1144°K (1600°F)

457-3086

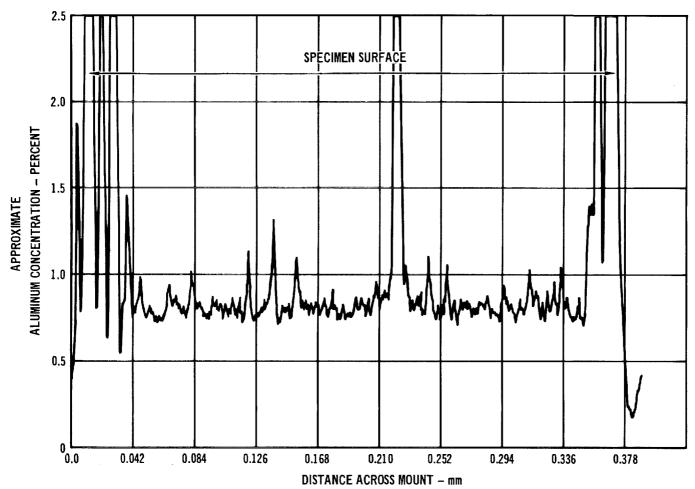
Figure 5-16 (b)

The environmental pressures used in the present study are shown in Figure 5-2 and are tabulated in Table 5-6 for various time intervals after the start of the entry cycle. Also included in this table are the temperature profiles for the lowest and highest temperature test cycle used in this study. It can be seen that, even for the high temperature cycle, the cooling rate is sufficiently fast that the





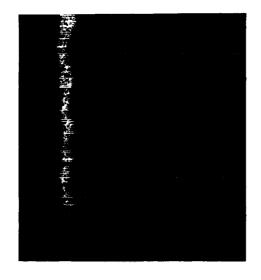
800X



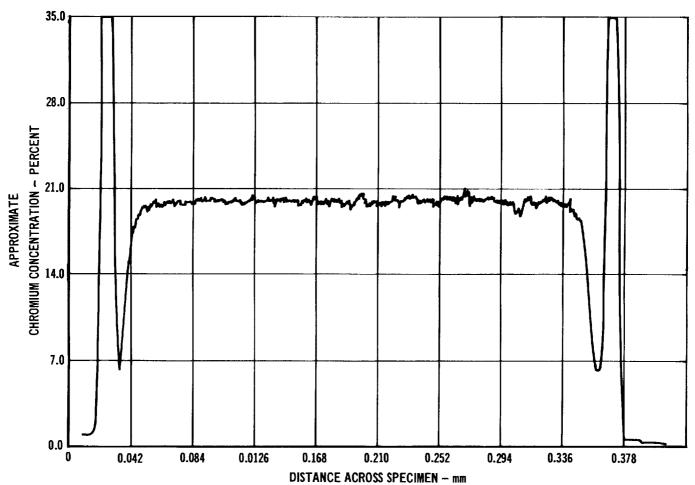
ALUMINUM X-RAY RASTER IMAGE AND MICROPROBE SCAN FOR RENE´41 SPECIMEN R-72 AFTER 50 SIMULATED ENTRY CYCLES AT 1144°K (1600°F), WITHOUT SALT COATING

457-3087

Figure 5-17(a)

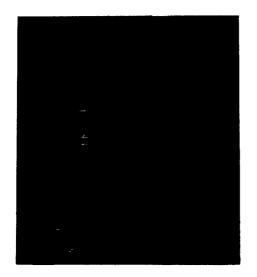


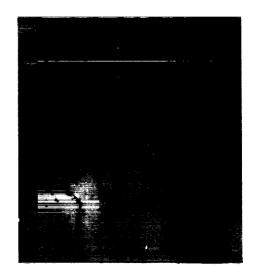


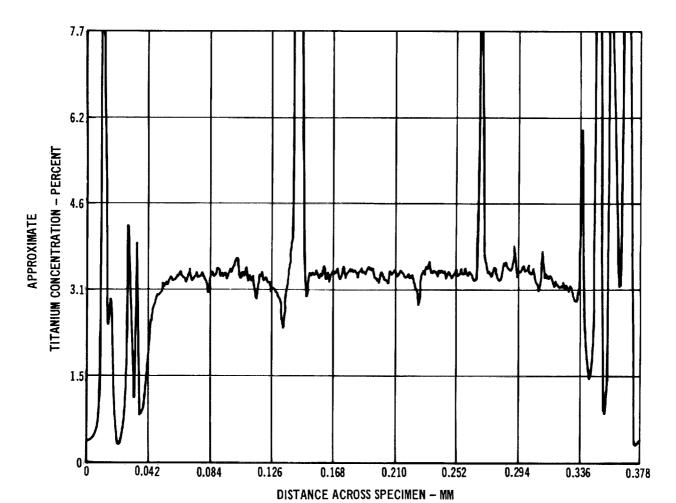


CHROMIUM X-RAY ROSTER IMAGE AND MICROPROBE SCANE FOR RENE'41 SPECIMEN R-72 (Continued)

Figure 5-17 (b)

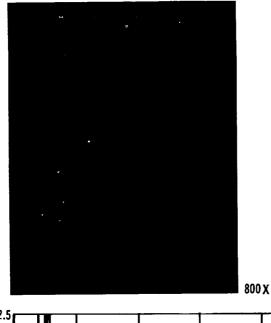


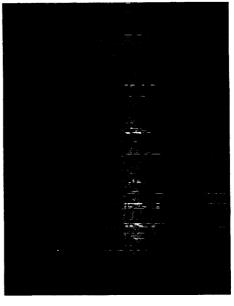




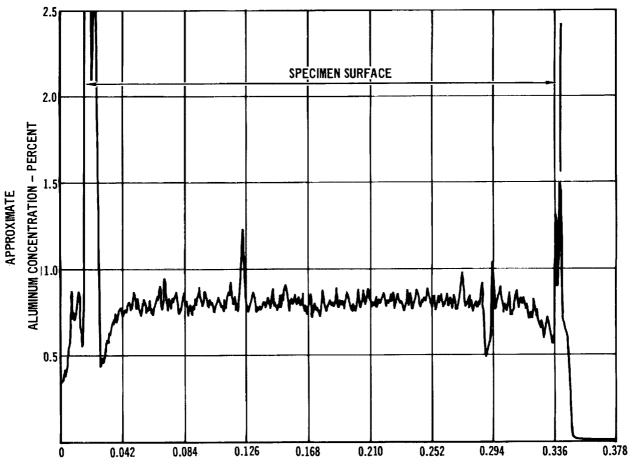
TITANIUM X-RAY ROSTER IMAGE AND MICROPROBE SCAN FOR RENE '41 SPECIMEN R-72 (Continued)

Figure 5-17 (c)





800X



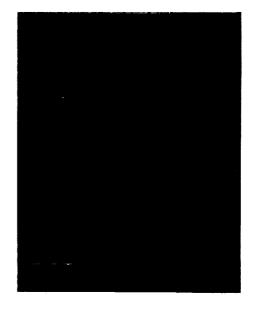
DISTANCE ACROSS MOUNT - mm

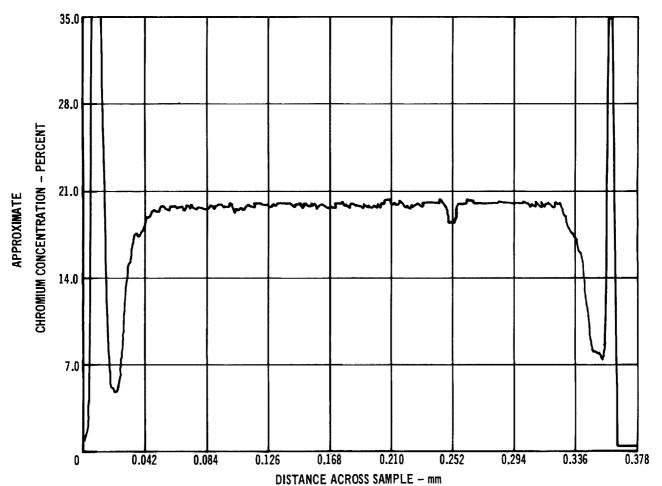
ALUMINUM X-RAY ROSTER IMAGE AND MICROPROBE SCAN FOR
RENE'41 SPECIMENT R-83 AFTER 50 SIMULATED ENTRY CYCLES
AT 1144°K (1600°F), WITH SALT COATING

457-3090

Figure 5-18 (a)



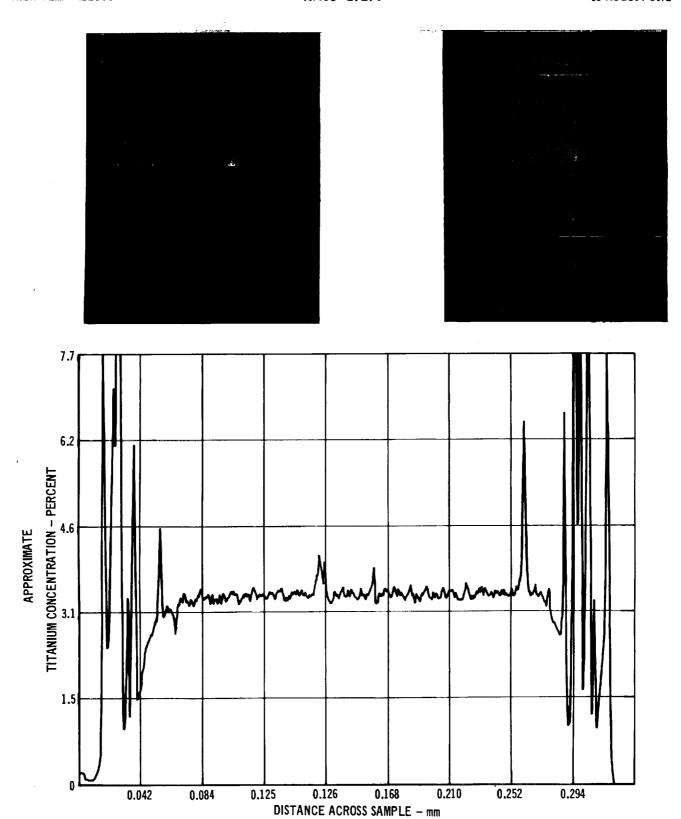




CHROMIUM X-RAY RASTER IMAGE AND MICROPROBE SCAN FOR RENE '41 SPECIMEN R-83 (Continued)

457 - 309 I

Figure 5-18 (b)



TITANIUM X-RAY RASTER IMAGE AND MICROPROBE SCAN FOR RENE '41 SPECIMEN R-83 (Continued)

Figure 5-18 (c)

partial pressure of oxygen at temperatures where a reaction might occur is much lower than the 1010 N/m^2 oxygen partial pressure of Reference 11. The results of this study, then, actually serve to confirm the dependence of the corrosion properties of these materials on salt coating thickness and the partial pressure of oxygen in the surrounding environment.

Table 5-6

TYPICAL TEMPERATURE/PRESSURE DATA FOR TWO DIFFERENT ENTRY CYCLES

TIME AFTER START OF CYCLE	CHAMBER PRESSURE				SPECIMEN TEMPERATURE			
	TOTAL		02 PARTIAL PRESSURE*		MAXIMUM T (CYCLE) = 811 ⁰ K (1000 ⁰ F)**		MAXIMUM T (CYCLE) = 1589°K (2400°F)**	
(SEC)	N/M ²	TORR	N/M²	TORR	οK	°F	οK	°F
0	0.665	0.005	0.133	0.001	422	300	489	420
600	1.33	0.01	0.266	0.002	811	1000	1589	2400
2100	399	3	92.6	0.696	811	1000	1589	2400
2600	1064	8	247	1.86	566	560	922	1200
3100	2000	15	463	3.48	472	390	616	650
3600	3330	25	771	5.80	422	300	489	420

^{*}CALCULATED ASSUMING THE COMPOSITION OF AIR TO BE 23.2 W/O O₂, 76.8 W/O N₂

^{**}THESE CYCLES, USED FOR INCONEL 718 AND FS-85/R-512E, REPRESENT THE LOWER AND UPPER BOUNDS OF THE TEMPERATURE CYCLE PARAMETERS USED IN THIS STUDY.

⁴⁵⁷⁻³⁰⁹³

^{5.6} Conclusions - The results of the simulated reentry testing indicate that the alloys studied are immune to the effects of hot salt corrosion or stress corrosion under the particular test conditions used. Although a salt glazing reaction did occur for the FS-85 alloy cycled to 1589°K (2400°F), attack was limited to the very surface of the R512E coating and did not penetrate to the underlying substrate during the test duration. The creep, crack propagation, and tensile properties of each alloy are insensitive to the presence of a salt coating. Furthermore, with the exception of the FS-85 alloy which becomes oxygen embrittled, the tensile properties of each material are unaffected by reentry simulation. Although microstructural changes do occur in most of the alloys studied as a result of reentry simulation, such changes are not related to the presence of a salt coating.

6.0 CONCLUSIONS

The work accomplished during the first year of this program demonstrates the excellent immunity of candidate metallic TPS alloys to corrosion and stress corrosion in both salt water and hot salt simulated reentry environments. The complete lack of any slow crack growth, degradation in tensile properties, or evidence of general or pitting corrosion after 60 days of alternate salt water immersion indicates that a thermal protection system fabricated from any of the six alloys tested is not likely to interact with any aqueous salt environments. Even after becoming coated with dry salt, such a TPS is also very likely to withstand a minimum of 50 reentry cycles without significant changes in the creep, crack propagation, and tensile properties of the materials relative to the uncoated condition.

In the case of the FS-85 alloy, even though limited glaze formation was observed on salt coated specimens cycled to 1589°K (2400°F), oxygen embrittlement caused by defects in the silicide coating is considered to exert greater influence on the selection of the alloy for this application.

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APPENDIX A

In order to ascertain what research on stress corrosion problems was being conducted, 23 individuals from 19 companies in the United States and Canada were contacted. These contacted by letter were:

- o E. S. Bartlett of Battelle Memorial Institute, Columbus, Ohio
- o B. R. Barrett of Cabot Corp., Stellite Division, Kokomo, Indiana
- o L. J. Klingler of Fansteel, Inc., Baltimore, Maryland
- o E. Keller of Convair Division, General Dynamics Corp., San Diego, Calif.
- o W. C. Hazel of General Electric Company, Cincinnati, Ohio
- o S. Moser of General Electric Company, Cincinnati, Ohio
- O R. J. Morris of International Nickel Company, Dayton, Ohio
- o F. K. Lampson of Marquardt Corp., Van Nuys, Calif.
- o H. J. Brown of Martin-Marietta Corp., Denver, Colorado
- o B. Hello of North American Rockwell, Space Division, Downey, Calif.
- o F. L. Ver Snyder of Pratt and Whitney Aircraft, E. Hartford, Conn.
- O J. B. Moore of Pratt and Whitney Aircraft, West Palm Beach, Fla.
- o M. A. Clegg of Sherritt Gordon Mines LTD., Fort Saskatchewan, Alberta
- o P. A. Bergman of Thompson Laboratories, Lynn, Mass.
- o L. W. Lherbier of Specialty Steel Division, Universal Cyclops Corp.,
 Bridgeville, Pa.
- O E. Urbas of Westinghouse Astronuclear Corp., Pittsburg, Pa.
- A sample of our letter, and the replying letters received, are included.

In addition, several other aerospace companies and organizations were contacted by telephone. They were:

- O B. F. Brown of Naval Research Lab., Washington, D.C.
- o S. Goldberg of NAVAIR, Washington, D.C.
- O R. Carter of Boeing Company, Seattle, Washington
- o C. Hartbower of Aerojet General, Sacramento, California
- o V. Dress of Lockheed

All 5 men replies that they had no active stress corrosion research or evaluation programs on high temperature alloys.

E. Urbas of Westinghouse Astronuclear Corp. responded by telephone. He informed us about work being done by Neil Pessell, of the Westinghouse Research and development Center, to develop a low cost iron-base alloy that resists corrosion in hot sea water. The program is sponsored by the Office of Saline Water, Department of the Interior, under contract number 14-01-0001-2299. Progress reports number 478, September 1969, and number 627, December 1970, were recommended for review.

Some experimental automobile emission control devices utilize high temperature alloys. Thinking that they may be experiencing corrosion of stress corrosion problems, we contacted the metallurgical department of General Motors Research Laboratories in Detroit. They told us that although they had experienced corrosion from sulfur-containing fuel products with turbine alloys and in reactors, they had not looked into the problem, and could be of little help for our program.

2 June 1971

SAMPLE LETTER

Dear Sir:

We are currently conducting a survey of past and present research into the corrosion and stress corrosion of high temperature alloys, under NASA Contract NAS-8-27270. Emphasis is placed on studies conducted on materials exposed for 30 days in 3 1/2% salt sea water and then tested at temperatures from RT to 2500°F. The alloys of particular interest are the superalloys: Inconel 718, Rene' 41, Hastelloy X, TDNiCr, L605, Haynes 188 and the columbium alloy FS-85.

We would like to ascertain what work your Company has done in this area or any other work that is going on outside of your Company. If you have any reports or papers pertinent to this subject, I would appreciate your sending copies of them to me by 18 June 1971.

Sincerely yours,

MCDONNELL DOUGLAS ASTRONAUTICS COMPANY-EAST

J. W. Davis

Dept. E457, Bldg. 106-3 Phone: (314) 232-6458

JWD:ch

THE INTERNATIONAL NICKEL COMPANY, INC.

ONE NEW YORK PLAZA . NEW YORK, N. Y. 10004

PRODUCT RESEARCH AND DEVELOPMENT

June 3, 1971

Mr. John W. Davis McDonnell Douglas Corporation Building 106 Department E 457 P.O. Box 516 St. Louis, Missouri 63166

Dear Mr. Davis:

Enclosed are two articles on the hot corrosion behavior of some of the alloys of interest to you for Space Shuttle application. If you have additional questions, please call me at (212) 742-4580.

Very truly yours,

D. H. Maxwell

Gas Turbine Alloys,

Section Manager

DHM: im

att.

Pratt & Whitney Aircraft DIVISION OF UNITED AIRCRAFT CORPORATION

June 8, 1971

Mr. J. W. Davis McDonnell Douglas Astronautics Company - East Saint Louis, Missouri 63166

Dear Mr. Davis:

Re your letter of 2 June 1971 concerning your work under NASA Contract JAS-8-27270, we have reviewed our work on corrosion and stress corrosion of high temperature alloys such as Hastelloy X and others and find that we do not have any data concerning exposure in 3 1/2% salt sea water. The single company which I can think of which might have done work in this area is the International Nickel Company.

I wish you well in your efforts on this program.

Sincerely,

PRATT & WHATHAY ALRORAD

F. L. VerSnyder

Manager

Materials Engineering and Research Laboratory

FLV/el



1020 WEST PARK AVENUE, KOKOMO, IND. 46901

TELEPHONE 317 - 457-8411

June 9, 1971

McDonnell Douglas Astronautics Company Saint Louis, Missouri 63166

Attention: Mr. J. W. Davis

Dept. E457, Bldg. 106-3

Dear John:

In reference to your letter of June 2 and our telephone conversation of June 7, we do not have any stress-corrosion data at elevated temperatures on any high-temperature superalloys as outlined in your letter. If any data comes to my attention, I will forward it to you.

As you requested, I am enclosing two copies of a report by Ed Wagoner on the physical metallurgy and mechanical properties of HASTELLOY alloy X. I am also enclosing four recent data sheets on HAYNES alloy No. 188 which might be of interest to you.

If you have any questions concerning this data, please let me know.

Very truly yours,

STELLITE DIVISION Cabot Corporation

E. E. Jenkins Market Development

EEJ/mk

Enclosures

PRODUCERS OF THE ABRASION-, CORROSION-, AND HIGH-TEMPERATURE-RESISTANT ALLOYS DISTINGUISHED BY THE TRADE MARKS
"HAYNES" . . . "HAYNES STELLITE" . . . "HASCROME" . . . "HASTELLOY" . . . "HAYSTELLITE" . . . "STELLITE" . . . "MULTIMET"

GENERAL DYNAMICS

Convair Division

Kearny Mesa Plant, P. O. Box 1128, San Diego, California 92112 · 714 - 277 - 8900 Procurement, P.O. Box 172 · Accounting, P.O. Box 1708
Lindbergh Field Plant, P.O. Box 1950, San Diego, California 92112 · 714 - 296 - 6611

In Reply Refer To 572-3-46
15 June 1971

Mr. J. W. Davis
Dept. E457, Bldg. 106-3
McDonnell Douglas Corporation
P. O. Box 516
Saint Louis, Missouri 63166

Dear Mr. Davis:

In regard to your letter of 10 June 1971 requesting data on superalloys exposed for 30 days in 3 $\frac{1}{2}\%$ salt sea water and then tested at various temperatures, we have none. It may be of interest to you to know that we are performing tests on the superalloys in the expected Space Shuttle environment. This will be available through NASA at a later date.

Sorry we are not able to be of assistance to you at this time.

Sincerely yours,

W. M. Sutherland

Test Lab Group Engineer

Mail Zone 572-30

Kearny Mesa Plant P. O. Box 1128

San Diego, California 92112

WMS:hv



RRITT GORDON MINES LIMITED / RESEARCH & DEVELOPMENT OFFICE AND

Fort Saskatchewan, Allerta, Canada/Telephone 403 543-69; (/Telex 03) 2299

14 June, 1971

Mr. J. W. Davis,
Department E457, Building 106-3
McDonnell Douglas Astronautics Company - East,
St. Louis, Missouri 63166
U.S.A.

Dear Mr. Davis:

Thank you for your letter of 7 June, 1971 enquiring after information on corrosion and stress corrosion testing.

The only work we are doing in this field is that on the oxidation resistance of our modified dispersion strenthened nickel-chromium alloys. The alloy composition is varied and testing is primarily evaluation of dynamic oxidation resistance in a burner rig, which includes hot corrosion testing with chloride contamination.

This work is supported by the Defence Research Board as Project XM36 and by the USAF under contract F33615-70-C-1199. The first annual Technical Report AFML-TR-70-247 was issued in October 1970 and we are pleased to enclose a copy herewith. The second annual report is due to be issued on the 30 September, 1971.

Yours sincerely,

Manager,

Di ...

Physical Metallurgy Research.

MAC/rs



AIRCRAFT

ENGINE

I GROUP

1000 WESTERN AVENUE, WEST LYNN, MASSACHUSETTS 01905 . . . TELEPHONE (617) 594-0100

cc: LP Jahnke, M-82 Evendale DW Puffer, 3-68 Lynn JS Mosier, N-127 Evendale

June 15, 1971

Mr. J. W. Davis McDonnell Douglas Astronautics Company-East Dept. E457, Bldg. 106-3 Saint Louis, Missouri 63166

Dear Mr. Davis:

Your letter of June 2 to Stan Mosier has been referred to me. Within General Electric's AEG, work on salt sea water corrosion effects such as you describe has been quite limited. We do not have available any reports suitable for distribution outside of the company. However, the following reports from the literature may be of help to you in your NASA program.

"Chloride Stress Corrosion Susceptibility of High Strength Stainless Steel, Titanium, and Superalloy Sheet", Douglas Aircraft Co., Air Force Report MLTD64-44, March 1964.

Raring, R.H., Freeman, J.W., Schultz, J.W., and Voorhees, H.R., "Progress Report of the NASA Special Committee on Materials Research for Supersonic Transports", NASA TN D-1798, May 1963.

Crossley, F.A., Reichel, C.J., and Simcoe, C.R., "The Determination of the Effects of Elevated Temperatures on the Stress Corrosion Behavior of Structural Materials", WADD TR60-191, May 1960.

I trust that this information will prove of value to you.

Very truly yours

C. F. Muench, Manager
Materials Evaluation
THOMSON LABORATORY
Mail Drop 36804 - Ext. 5230
hk

Defense Metals Information Center

Battelle Memorial Institute
505 KING AVENUE
COLUMBUS, OHIO 43201
AREA CODE 614, TELEPHONE 299-3151

June 18, 1971

Mr. J. W. Davis
McDonnell Douglas Astronautics
Company-East
Department E457, Building 106-3
St. Louis, Missouri 63166

Dear Mr. Davis:

Your recent request to Mr. Ed Bartlett for information on the corrosion and stress corrosion of high-temperature alloys in salt sea water has been referred to me for reply.

Enclosed are photocopies of selected extract cards which deal with this subject. There were varying degrees of information among the specific alloys you are interested in, ranging from a good deal of information on Inconel 718 to virtually none on Haynes 188.

If we can be of further service, please do not hesitate to contact us.

Sincerely,

Ronald J. O'Brien

RJO:rac

Enc.



Space Division North American Rockwell

12214 Lakewood Boulevard Downey, California 90241

July 8, 1971

71MA4151

McDonnell Douglas Astronautics Company - East Saint Louis, Missouri 63166

Attention:

J. W. Davis - Dept. E457, Bldg. 106-2

Reference:

Letter Dated 15 June 1971

Dear Mr. Davis:

In line with our continuing interest in the behavior of materials, we have been undertaking some exploratory studies of the stress corrosion of high temperature alloys. These studies are exploratory in nature and designed only to identify potential problem areas. No reports or papers have been produced to date, but we shall be pleased to add your name to our distribution list when such documents are released.

Very truly yours,

NORTH AMERICAN ROCKWELL CORPORATION

B. Hello

Vice President and General Manager

Space Shuttle Program

Space Division

2744MA



UNIVERSAL-CYCLOPS SPECIALTY STEEL DIVISION

BRIDGEVILLE, PENNSYLVANIA 15017 | (412) 221-8000

RESEARCH AND DEVELOPMENT DEPARTMENT

July 27, 1971

Mr. J. W. David
Dept. E457, Bldg. 106-3
McDonnell Douglas Astronautics Co.-East
Saint Louis, Missouri 63166

Dear John:

As I mentioned to you some time ago on the telephone, we have not done any work involving the stress corrosion properties of high temperature alloys. Our work in the stress corrosion field has been minimal and confined to the ordinary austenitic stainless steels.

The work you are conducting under NASA Contract NAS-8-27270 sounds very interesting. I would appreciate any data you could give me on this program when it is released for publication.

Sincerely,

UNIVERSAL-CYCLOPS SPECIALTY STEEL DIVISION

L. W. Lherbier

Manager - Pesearch

LWL/dmr

MARTIN MARIETTA CORPORATION

DENVER DIVISION

POST OFFICE BOX 179, DENVER, COLORADO 80201 TELEPHONE (303) 794-5211

29 July 1971

McDonnell Douglas Astronautics Company-East St. Louis, Missouri 63166

Att: J. W. Davis
Department E457
Bldg. 106-3

Dear Mr. Davis:

In reply to your letter dated 2 June 1971, the Martin Marietta Corporation-Aerospace Division has no data relative to the corrosion resistance/susceptibility of superalloys, or refractory alloys, e.g., Inconel 718, Rene' 41, Hastelloy X, TDNiCr, L605, FS-85, etc., with 3½% salt solutions and exposed to temperatures ranging from R. T. to 2500°F.

With respect to work going on outside of our company, there are numerous reports concerning the above alloys in salt environments (mostly at ambient temperatures), but copies are not available from MMC. Better sources of these data would be the manufacturers of the specific alloys, International Nickel Corporation, Battelle Memorial Institute, the Boeing Corporation, and NASA-Lewis Research Center.

Most of our past work and literature searches have been oriented to corrosion susceptibility of non-ferrous and low temperature ferrous alloys in corrosive environments.

Sincerely,

Howard J. Brown

Mechanical Materials Engineering

/ra

THE

COMPANY 16555 SATICOY STREET • VAN NUYS, CALIFORNIA 91409 • TELEPHONE (213) 781 · 2121 • TWX 910 · 495 · 1710 • CABLE MARQCOR

August 6, 1971

McDonnell-Douglas Astronautic Company-East Dept. E457, Bldg. 106-3 Saint Louis, Missouri 63166

ATTENTION: Mr. John W. Davis

REFERENCE: Letter from J.W. Davis to F.K. Lampson dated 27

May, 1971.

Gentlemen:

We must first apologize for the delay in replying to your referenced letter, but plead the extreme pressures of business for our neglect. With regard to the request for data on several materials of construction, we have conducted a very limited amount of testing which could even be peripherally related to your request. Our testing in 3½% Salt was limited to 18% Ni. 250 and 18% Ni. 300 Maraging Steel. We tested none of the materials noted in your letter.

In the hopes that it might prove to be of some benefit in your evaluation, we are enclosing a copy of our document #S-962 (MPM 14.279) entitled:

> STRESS CORROSION EVALUATION OF 250 AND 300 GRADE 18% NICKEL MARAGING STEEL IN A 3-1/2% SALT SOLUTION INTERIM REPORT

> > Ву

E. E. Ritchie

Very truly yours,

THE MARQUARDT COMPANY CCI Aerospace Corporation

F.K. Lampson

Manager, Materials & Processes

33/L15

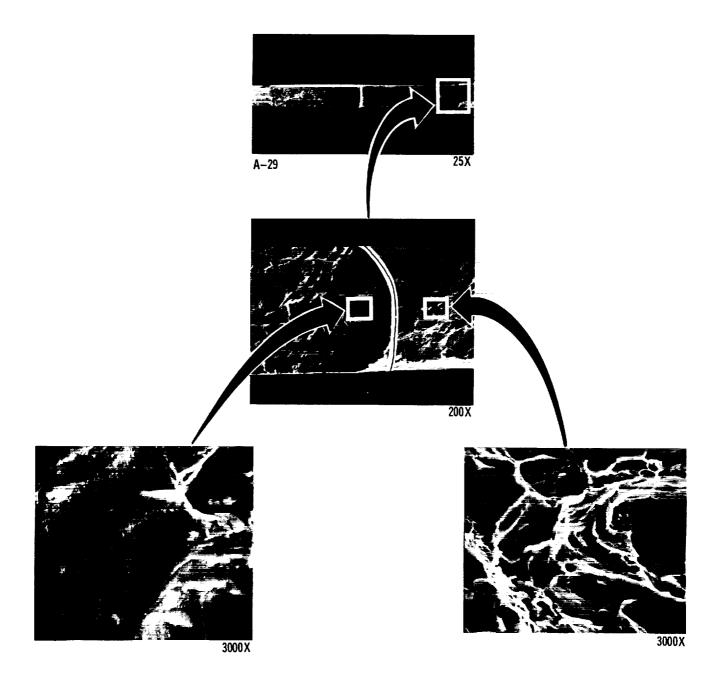
FKL/ff

encl:

4 Division of CCI Corporation - An Equal Opportunity Employer

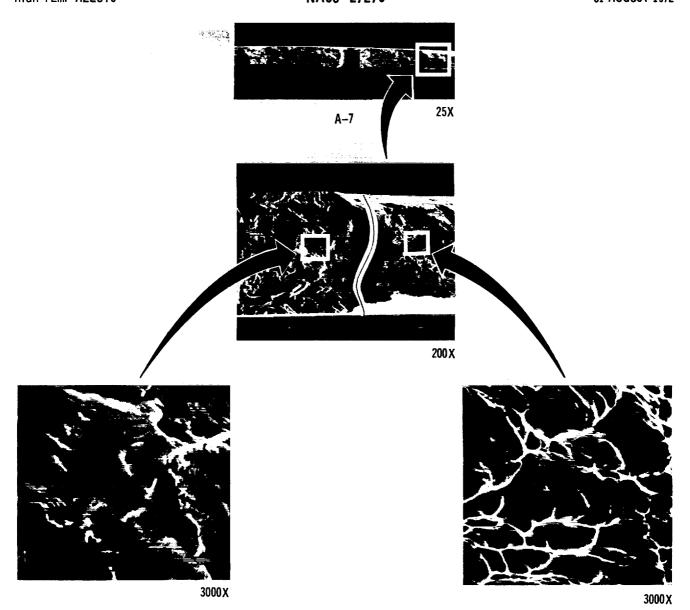
APPENDIX B

The fracture surfaces of tensile specimens subjected to 50 simulated entry cycles prior to room temperature testing are presented in the following photographs. Both salt coated and uncoated specimens material/test condition were examined and photographed with the Scanning Electron Microscope. It should be noted that fracture surfaces of the salt coated specimens are indistinguishable from their uncoated control specimens.



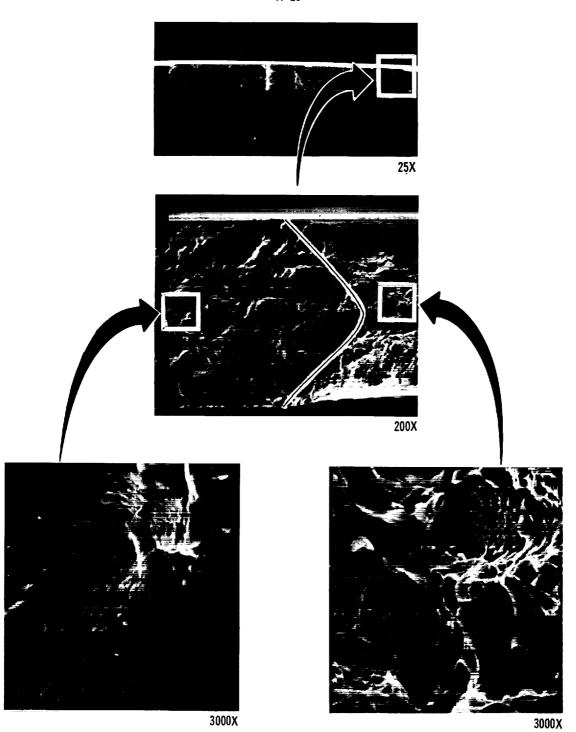
INCONEL 718 AFTER 50 REENTRY CYCLES AT 8110K (10000F) - SALT COATED

457-3094



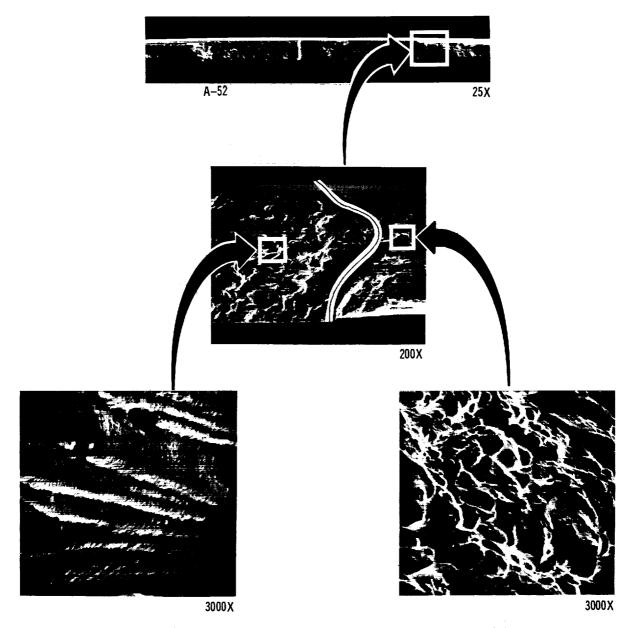
INCONEL 718 AFTER 50 REENTRY CYCLES AT 811°K (1000°F) - NOT SALT COATED

A-28



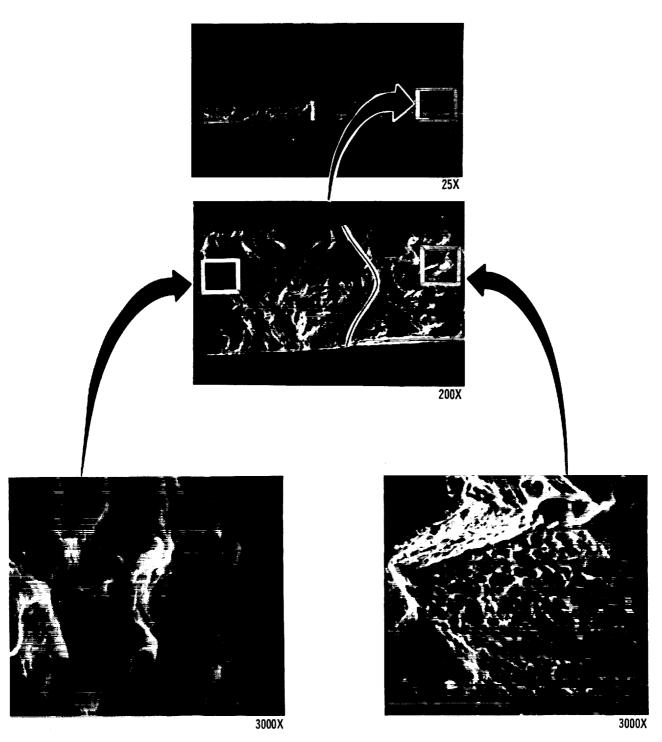
INCONEL 718 SPECIMEN AFTER 50 REENTRY CYCLES AT 922°K (1200°F) – SALT COATED

457-3096



INCONEL 718 AFTER 50 REENTRY CYCLES AT 922°K (1200°F) - NOT SALT COATED

R-9



RENE'41 SPECIMEN AFTER 50 REENTRY CYCLES AT 1033°K (1400°F) — SALT COATED

R-15 25X 200X

RENE '41 SPECIMEN AFTER 50 REENTRY CYCLES AT 1033°K (1400°F)
- NOT SALT COATED

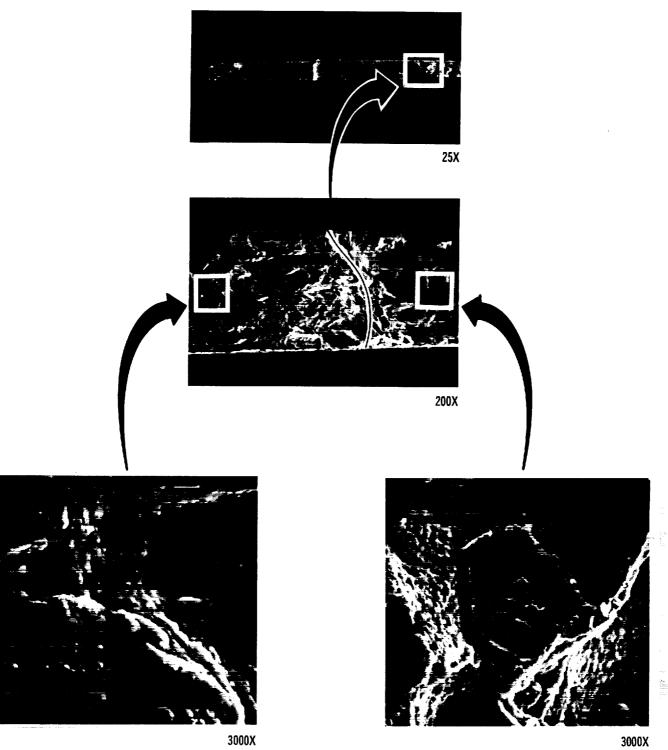
457-3099

Figure B-6

3000X

3000X

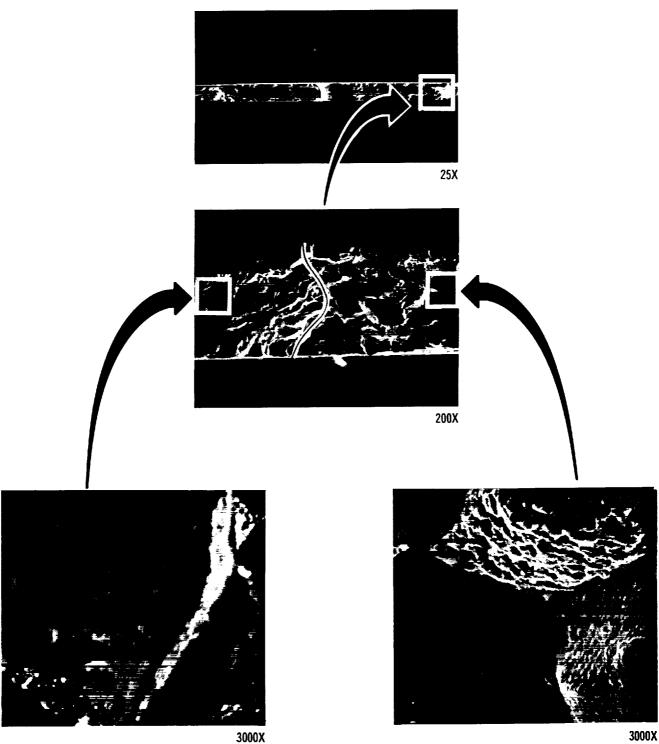
R-12



RENE 41 SPECIMEN AFTER 50 REENTRY CYCLES AT 1144°K (1600°F) – SALT COATED

457-3100

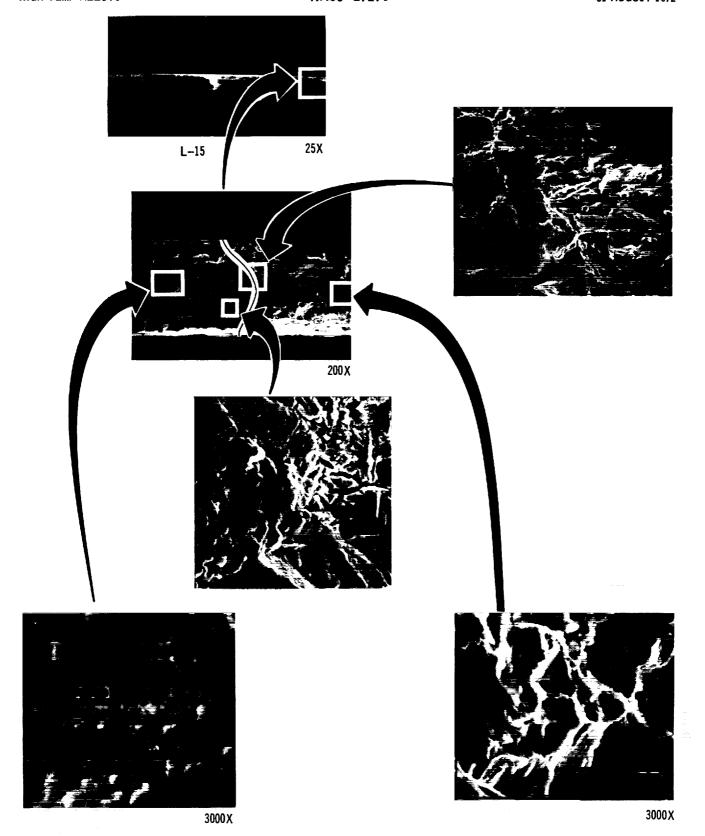
R-16



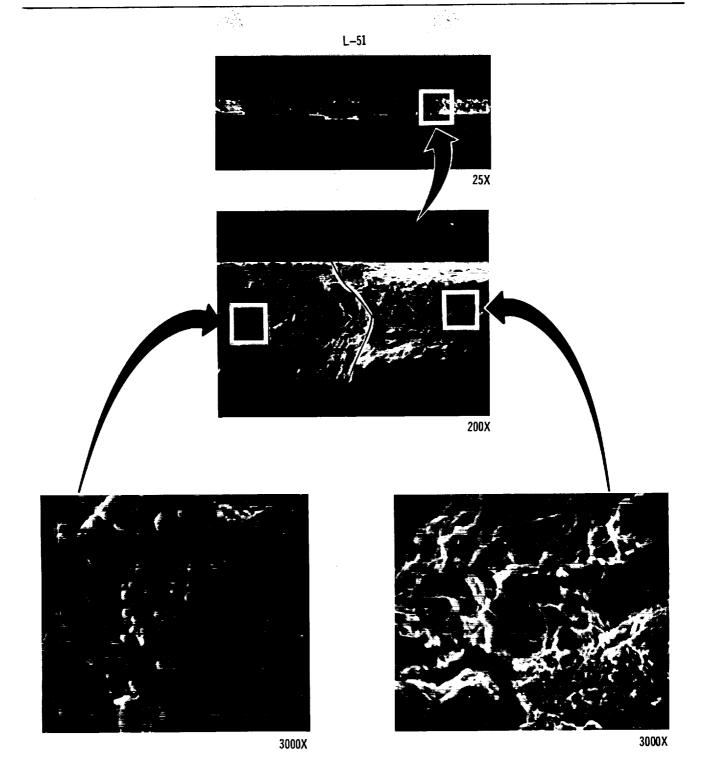
RENE 41 SPECIMEN AFTER 50 REENTRY CYCLES AT 1144°K (1600°F) - NOT SALT COATED

457-3101

457-3102

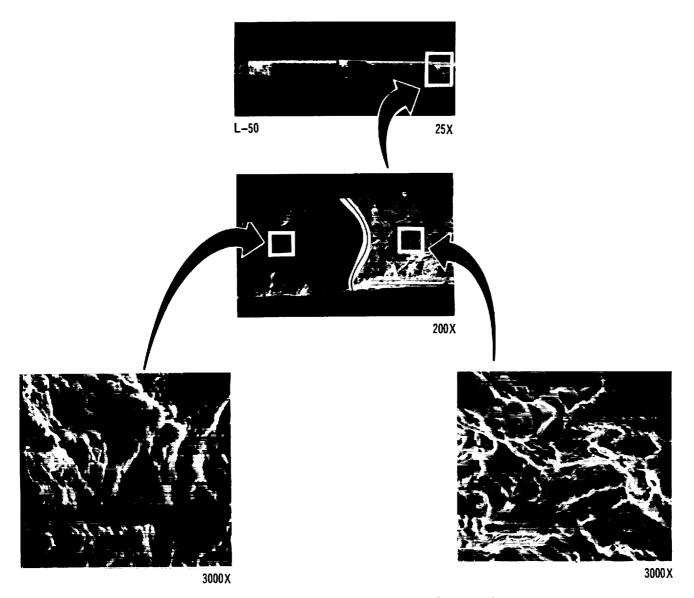


L605 SPECIMEN AFTER 50 REENTRY CYCLES AT 1144°K (1600°F) - SALT COATED Figure B-9

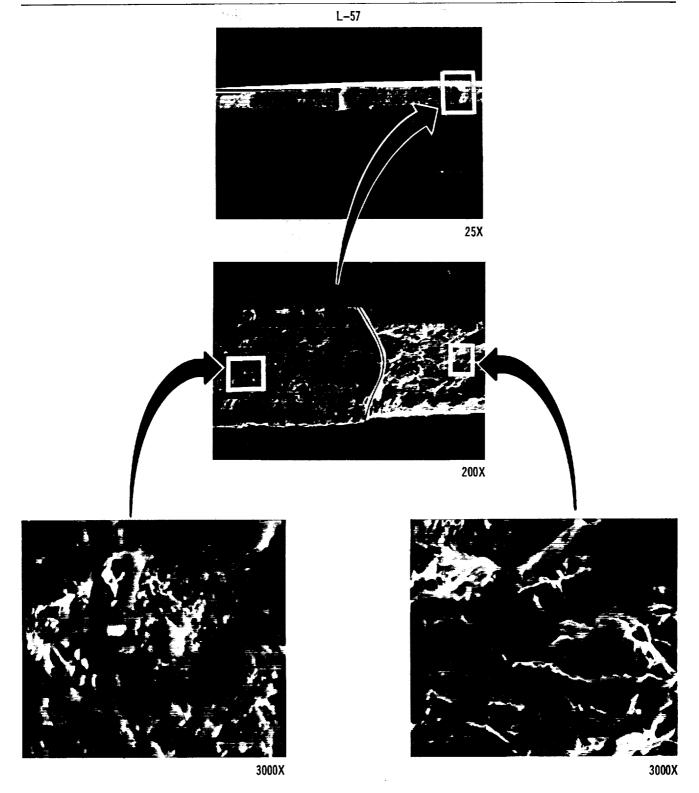


L605 SPECIMEN AFTER 50 REENTRY CYCLES AT 1144°K (1600°F)
- NOT SALT COATED

457-3103



L605 SPECIMEN AFTER 50 REENTRY CYCLES AT 12550K (18000F) - SALT COATED



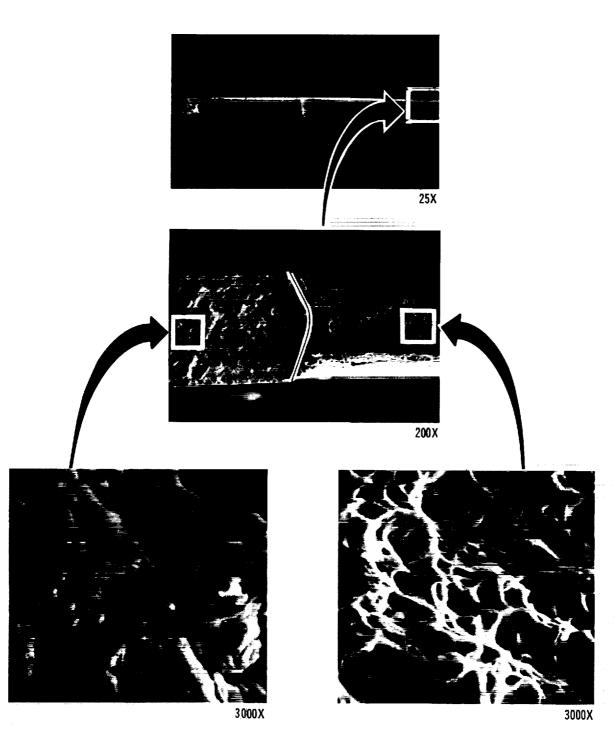
L605 SPECIMEN AFTER 50 REENTRY CYCLES AT 1255°K (1800°F)

- NOT SALT COATED

Figure

457-3105

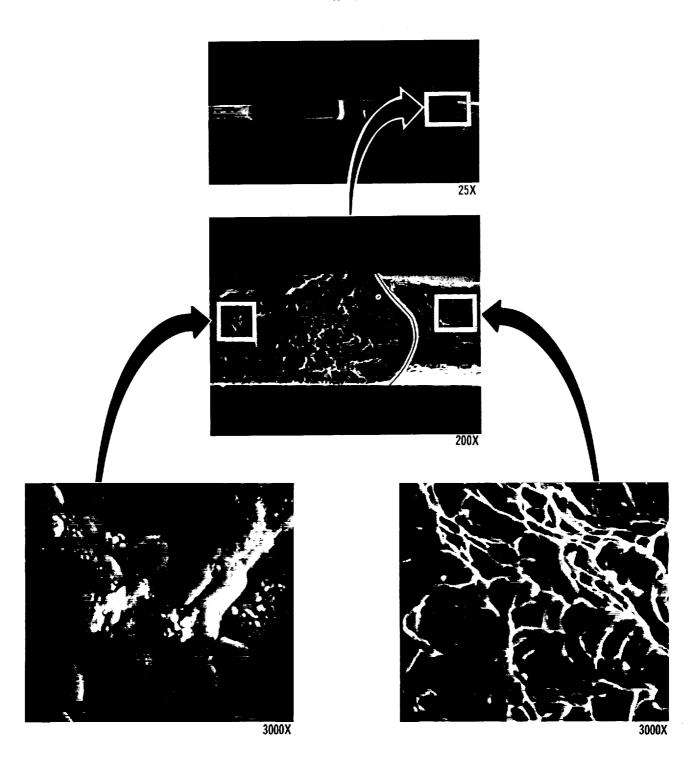
X-21



HASTELLOY X SPECIMEN AFTER 50 REENTRY CYCLES AT 1255°K (1800°F) – SALT COATED

457-3106

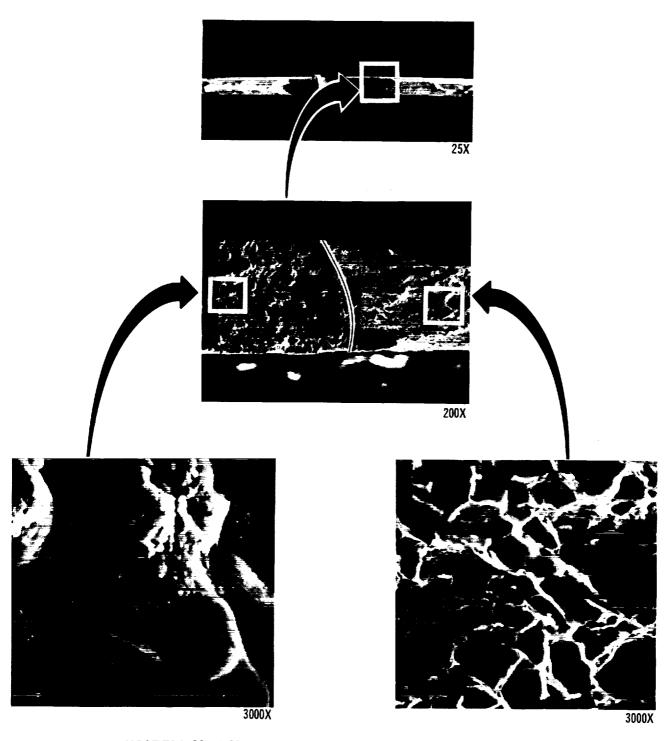
X-15



HASTELLOY X SPECIMEN AFTER 50 REENTRY CYCLES AT 1255⁰K (1800⁰F) – NOT SALT COATED Figure B-14

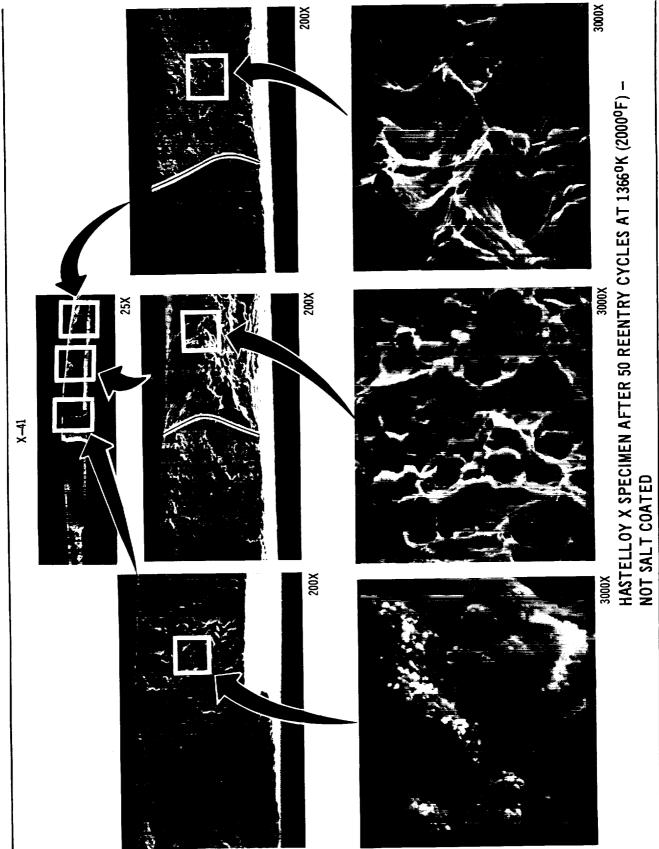
457-3107

X-28



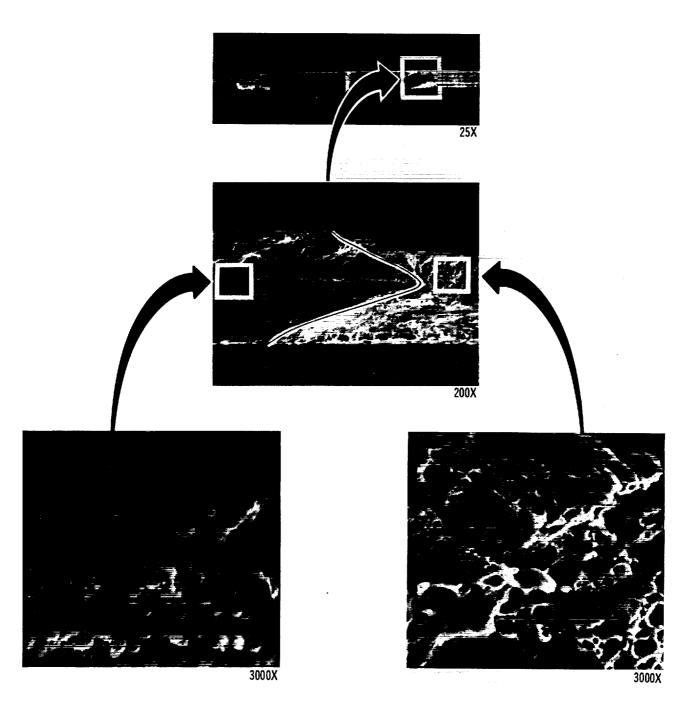
HASTELLOY X SPECIMEN AFTER 50 REENTRY CYCLES AT 1366°K (2000°F) — SALT COATED

457-3108



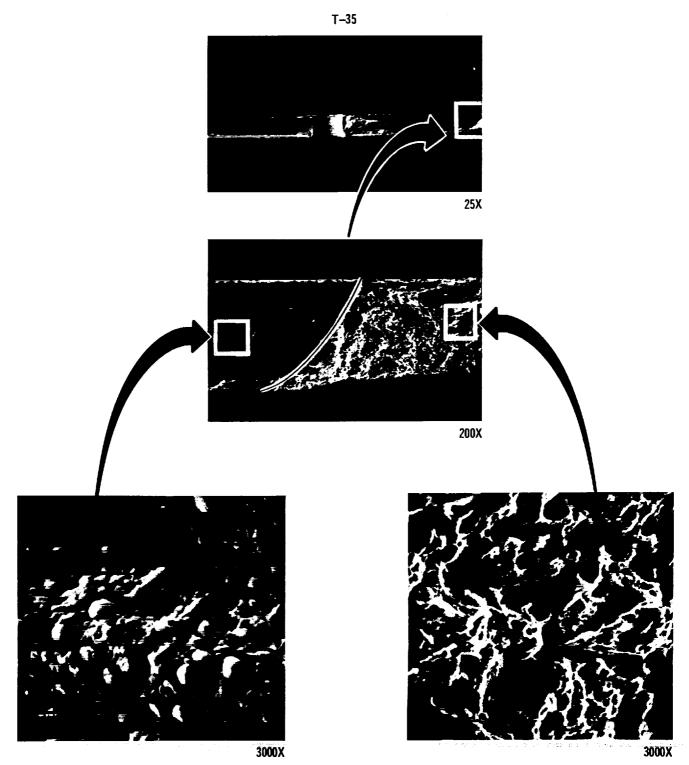
B-17

T-22



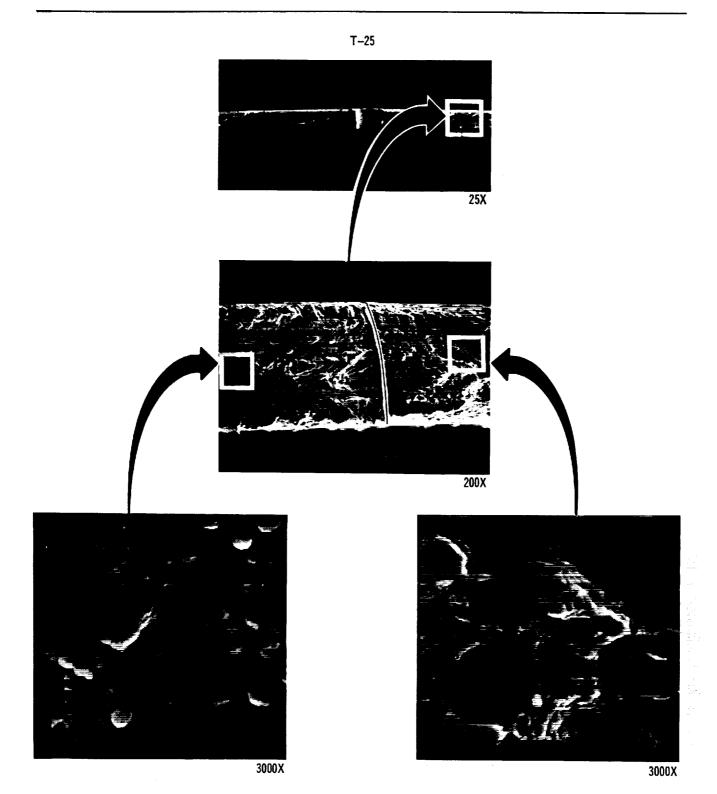
TD Ni Cr SPECIMEN AFTER 50 REENTRY CYCLES AT 1366°K (2000°F) — SALT COATED

457-3110



28 TD Ni Cr SPECIMEN AFTER 50 REENTRY CYCLES AT 1366°K (2000°F) - NOT SALT COATED

457~3111



TD Ni Cr SPECIMEN AFTER 50 REENTRY CYCLES AT 1477°K (2200°F) — SALT COATED

T-53 25X 200X

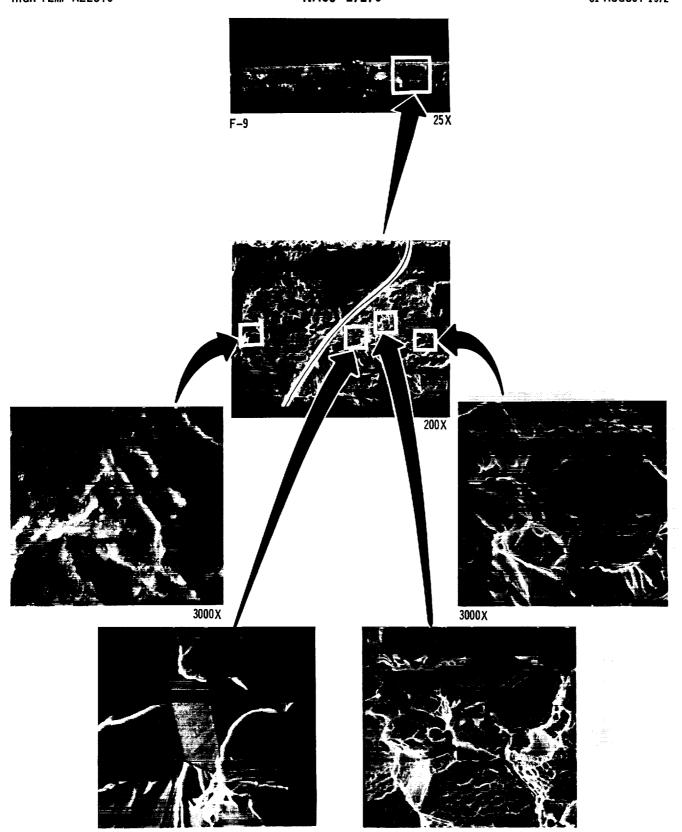
TD Ni Cr SPECIMEN AFTER 50 REENTRY CYCLES AT 1477°K (2200°F) — NOT SALT COATED

457-3113

Figure B-20

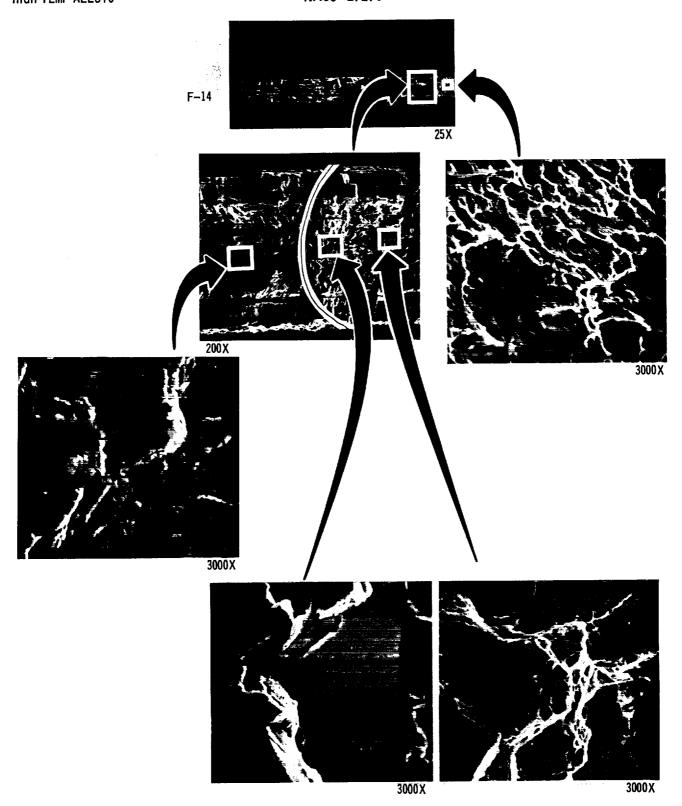
3000X

3000X

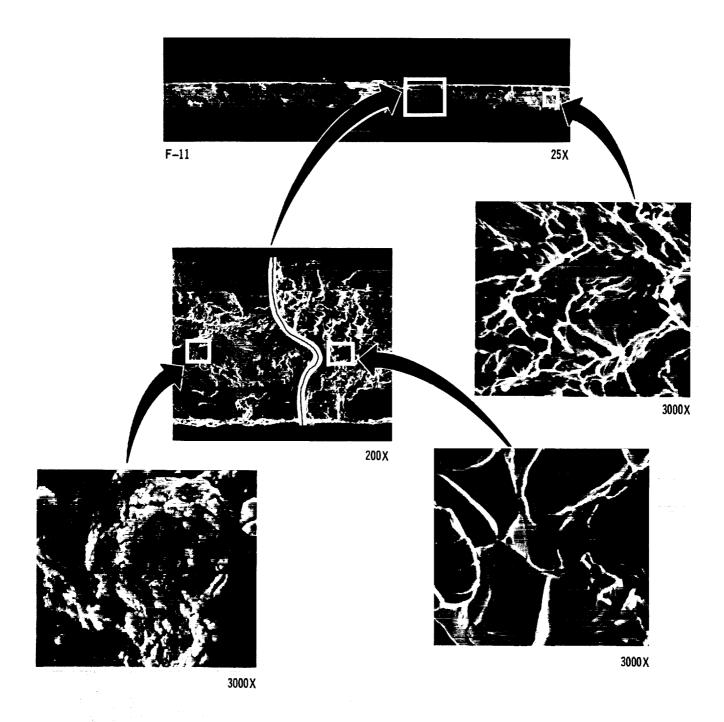


FS85 SPECIMEN AFTER 50 REENTRY CYCLES AT 1477°K (2200°F) - SALT COATED

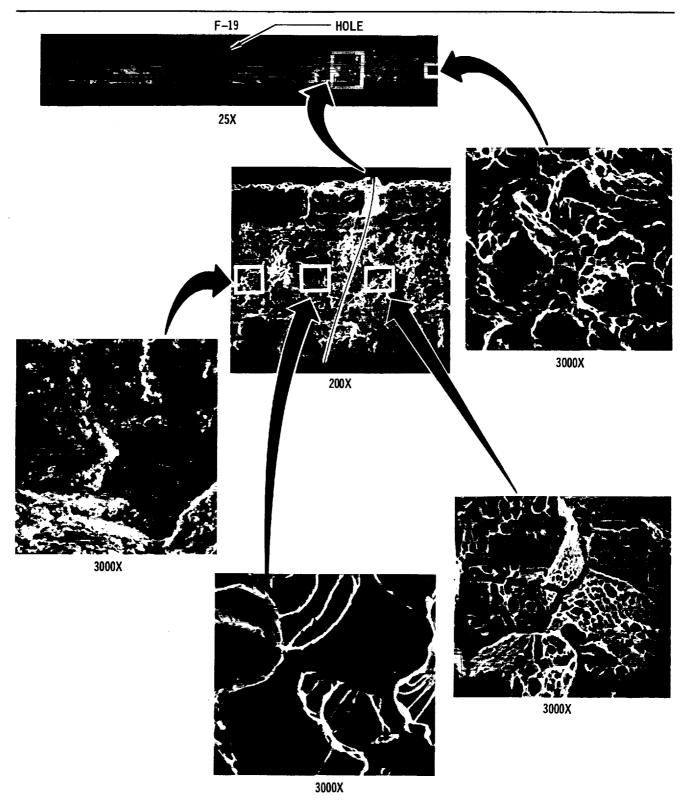
457 – 3114



FS85 SPECIMEN AFTER 50 REENTRY CYCLES AT 1477°K (2200°F) - NOT SALT COATED



FS85 SPECIMEN AFTER 50 REENTRY CYCLES AT 15890K (24000F) - SALT COATED



FS-85 SPECIMEN AFTER 50 REENTRY CYCLES AT 1589°K (2400°F)
- NOT SALT COATED
Figure

457-3117

•

APPENDIX C

TO CONVERT FROM	<u>TO</u>	MULTIPLY BY
Fahrenheit	kelvin	$t_{K} = (5/9)(t_{F} + 459.67)$
foot	meter	3.048×10^{-1}
inch	millimeter	$2.54 \times 10^{+1}$
lbf/inch ² (psi)	newton/meter ²	6.895×10^3
nautical mile	meter	1.852×10^3
torr (mm Hg)	newton/meter ²	1.33×10^2
Btu/ft ² second	watt/meter ²	1.135 x 10 ⁴

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